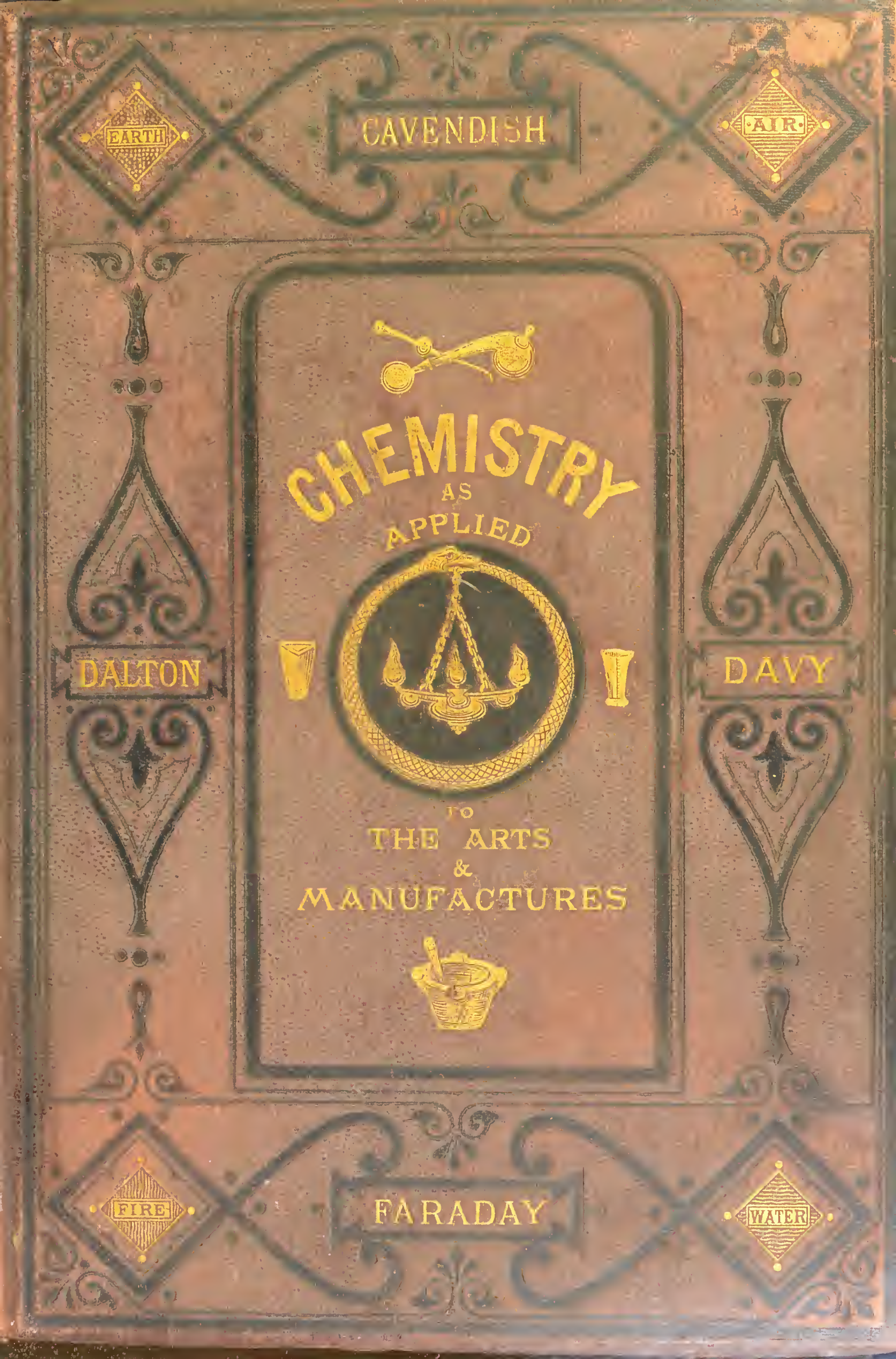

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CHEMISTRY AS APPLIED TO THE ARTS & MANUFACTURES

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CHEMISTRY,

THEORETICAL, PRACTICAL, AND ANALYTICAL,

AS APPLIED TO THE

ARTS AND MANUFACTURES.

BY

WRITERS OF EMINENCE.

ESTABLISHED 1803.

VOL. I.

LONDON:
WILLIAM MACKENZIE, 69 LUDGATE HILL, E.C.
DUBLIN, EDINBURGH, AND GLASGOW.

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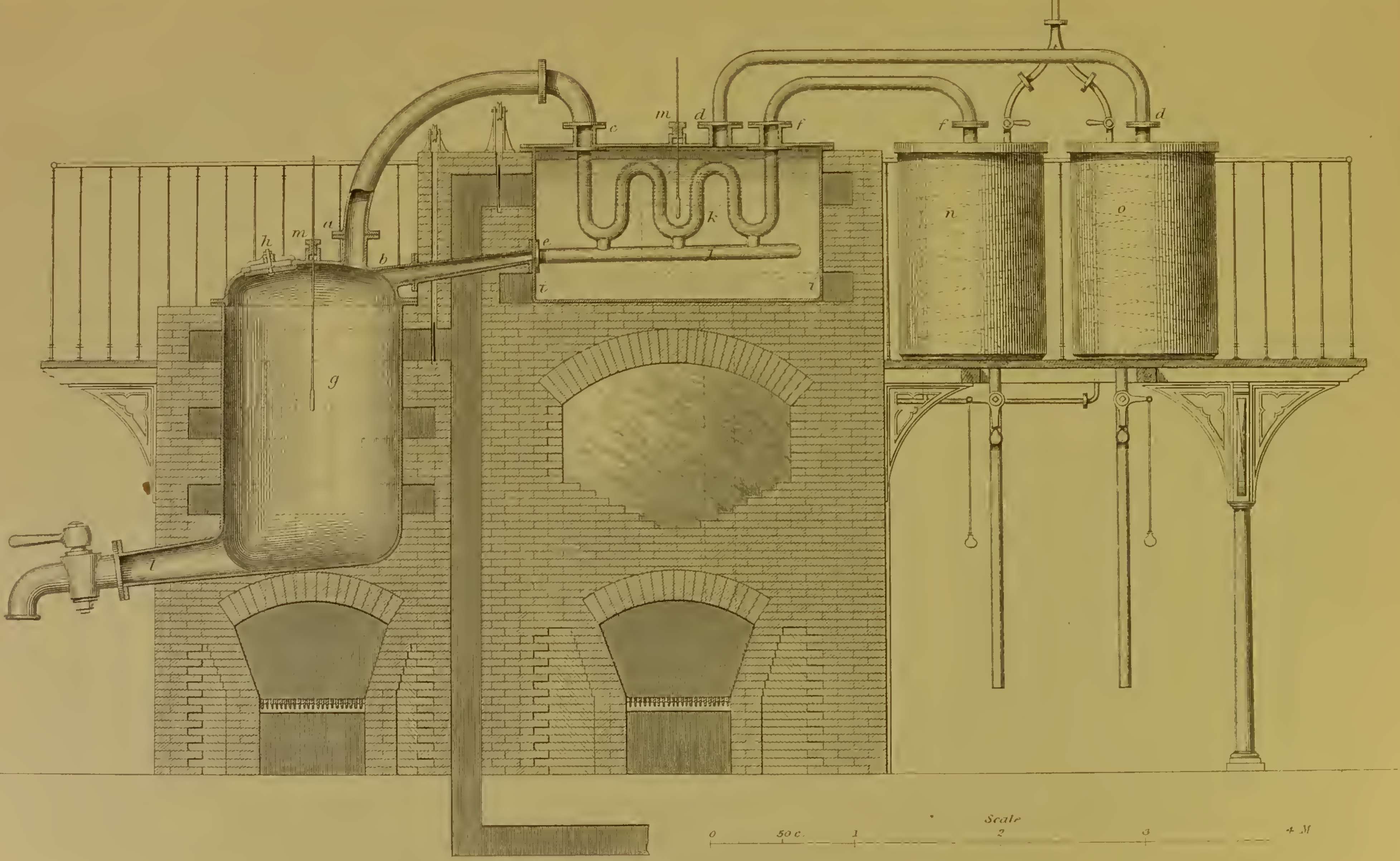
APPARATUS FOR THE FRACTIONAL DISTILLATION OF ANILINE OILS.



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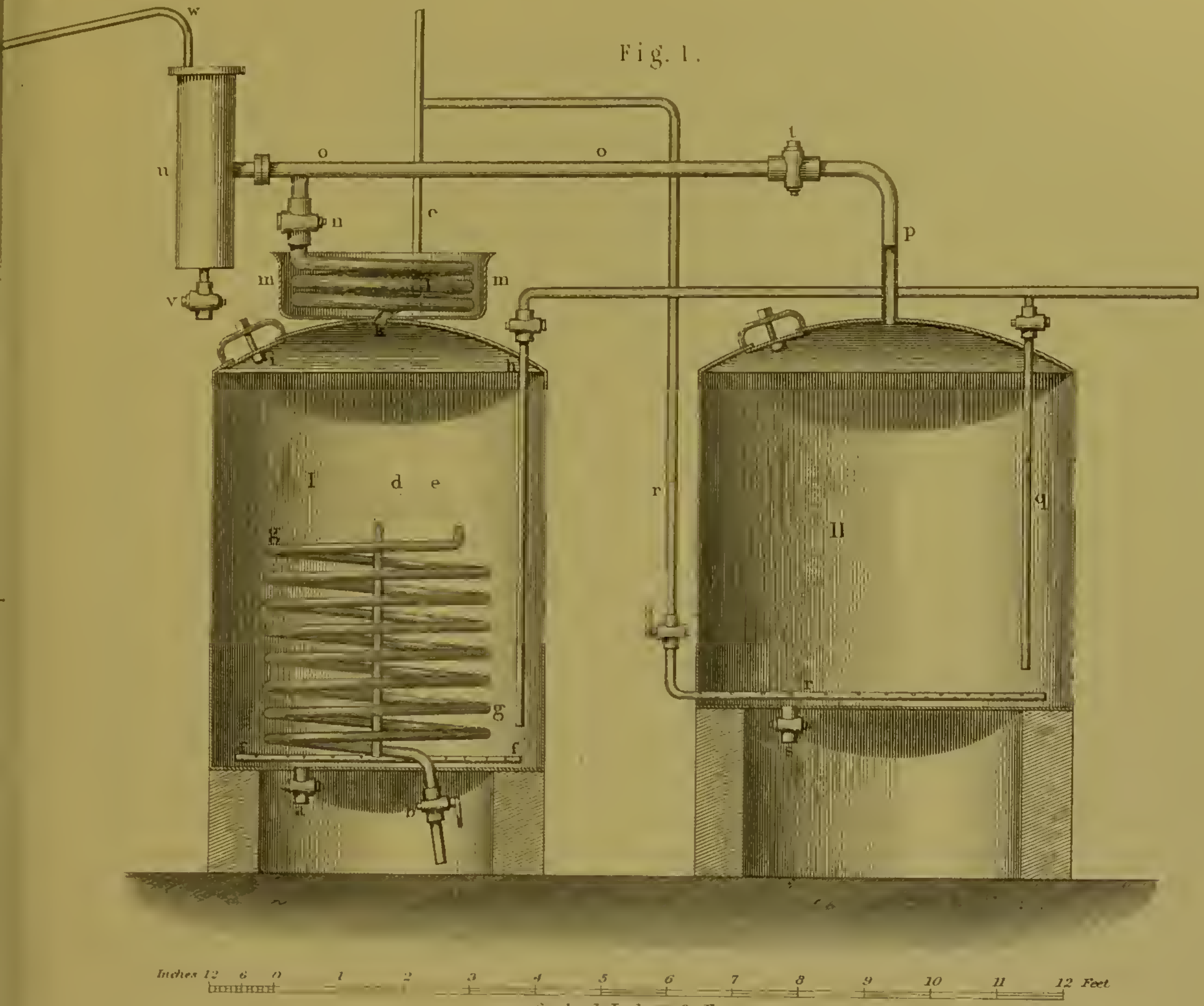
BENZOL.

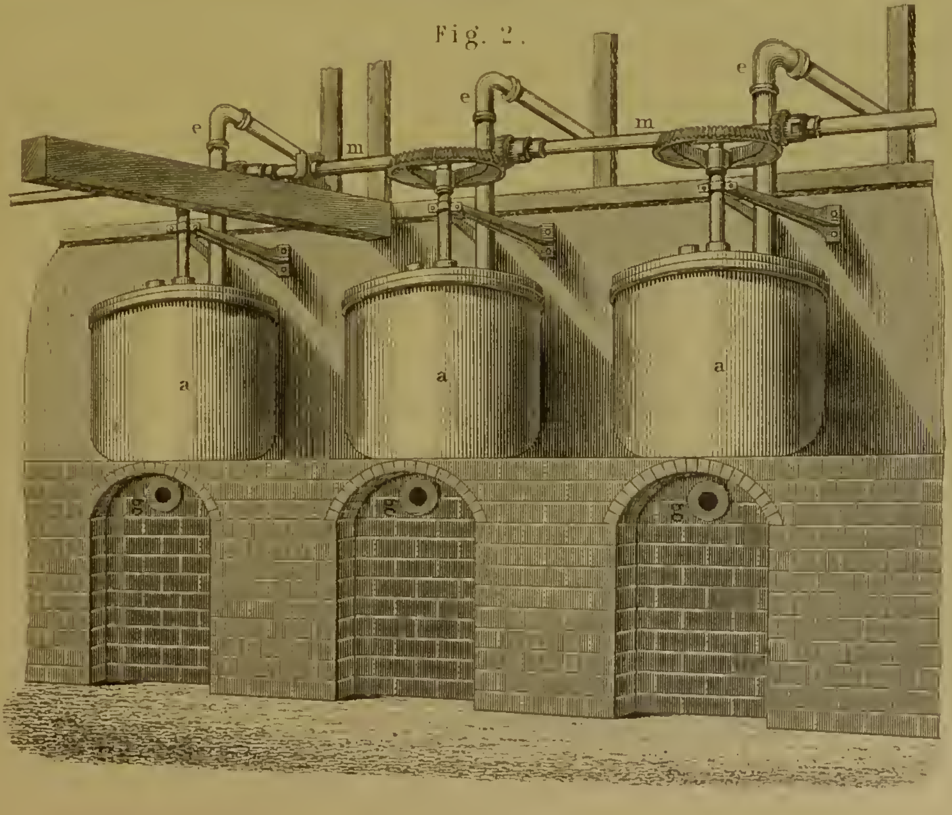
STILLS.

Fig. 1.



Scale 1 Inch = 3 Feet

Fig. 2.



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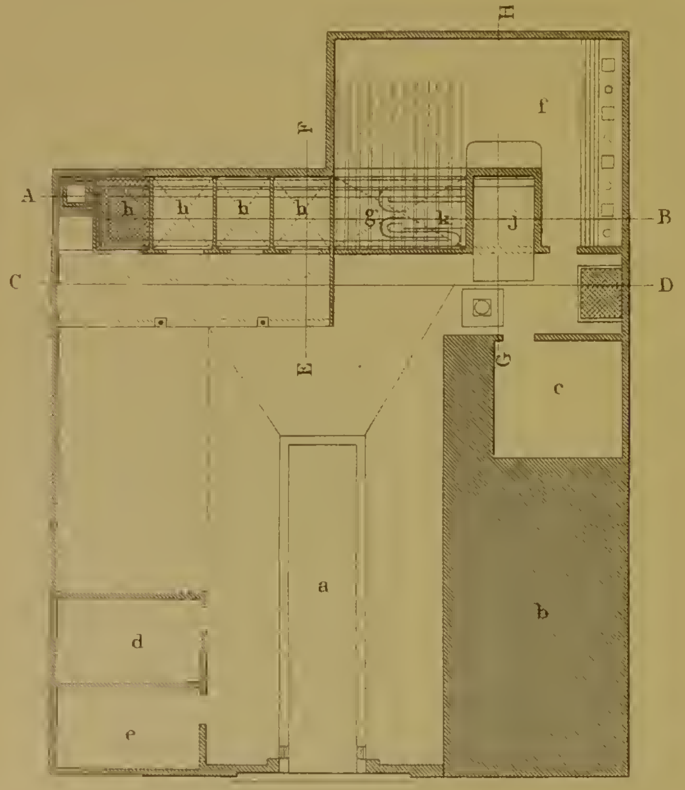
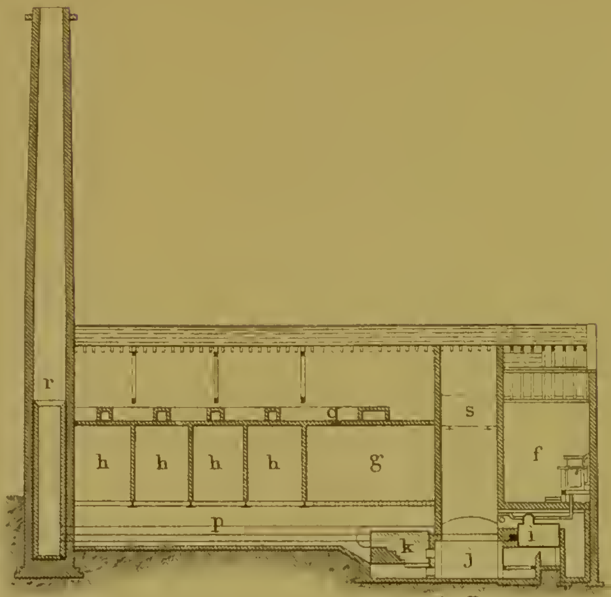
DISINFECTING APPARATUS, AT NEW BIRD Sᵀ LIVERPOOL.

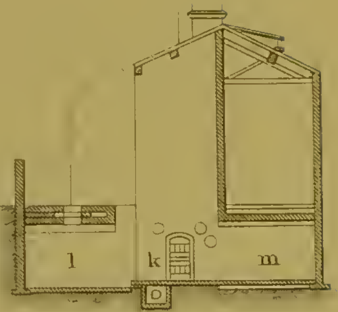


New Bird Sᵗ

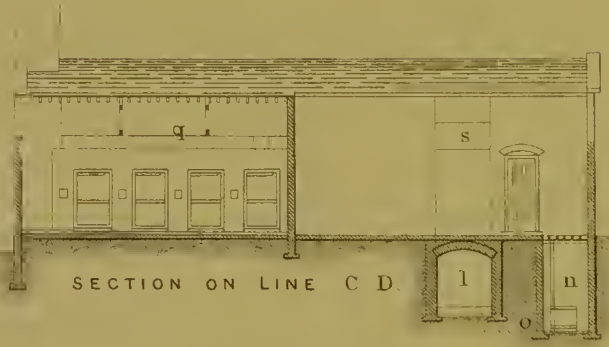
GROUND PLAN



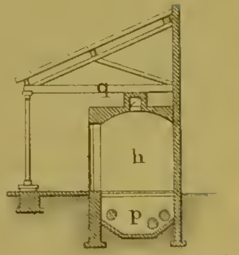
SECTION ON LINE A.B.



SECTION ON LINE G.H.



SECTION ON LINE C D.



SECTION ON LINE E.F.

SCALE OF FEET

10 5 0 10 20 30 40 50 60 70 80 90

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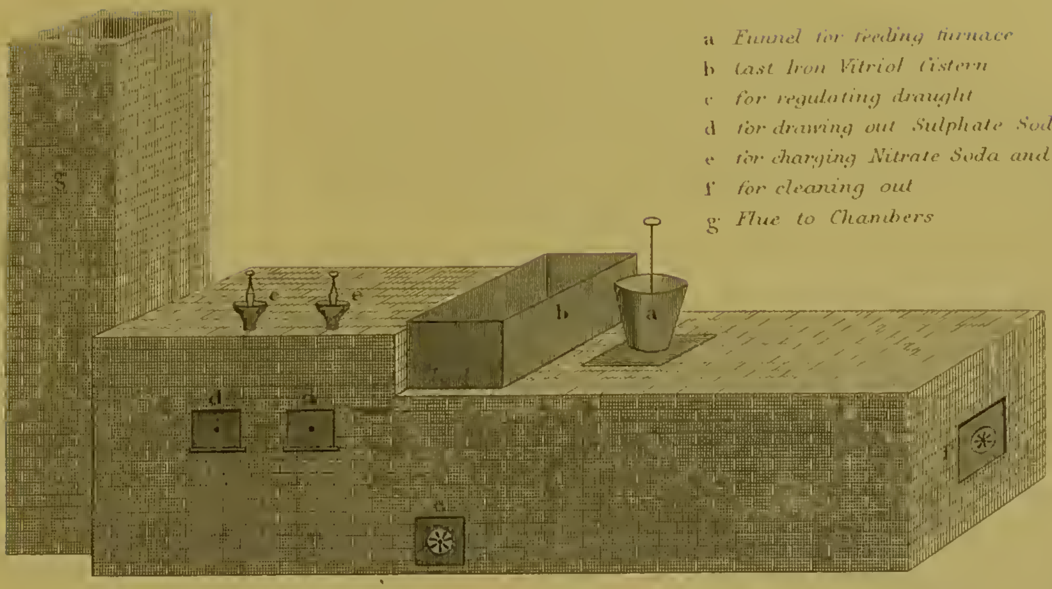
SULPHURIC ACID.

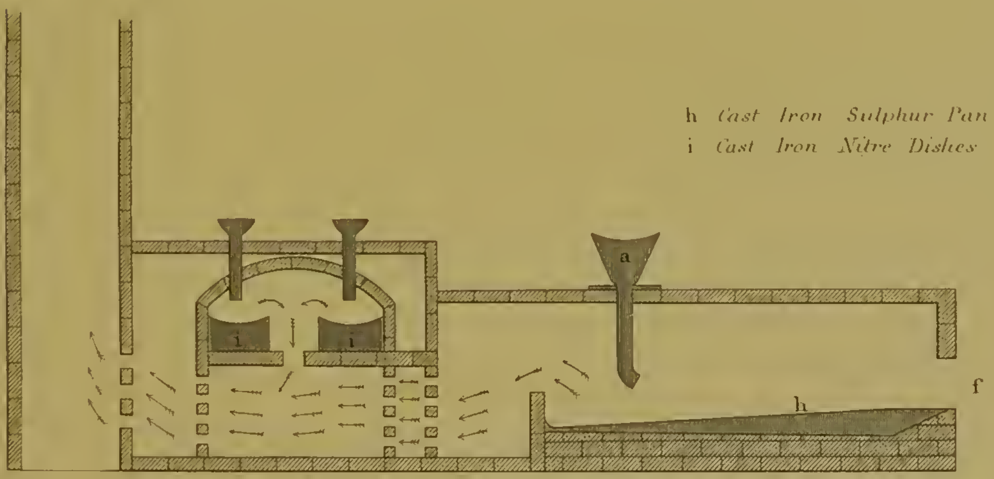
PLATE I.

PYRITES KILN.



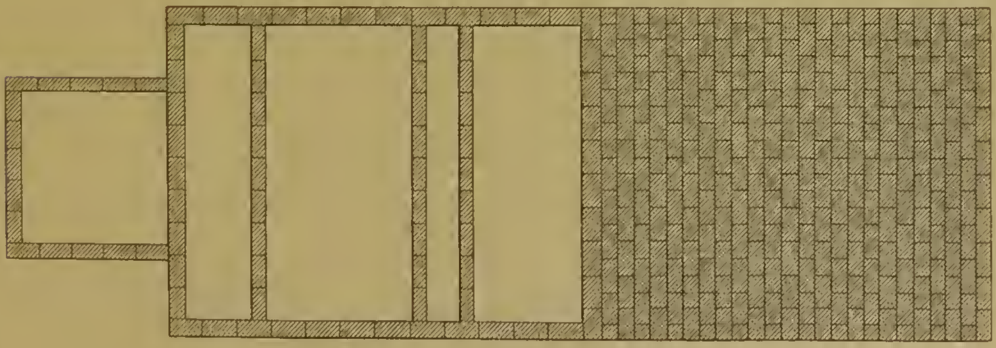
a *Funnel for feeding furnace*
b *Cast Iron Vitriol Cistern*
c *for regulating draught*
d *for drawing out Sulphate Soda*
e *for charging Nitrate Soda and Vitriol*
f *for cleaning out*
g *Flue to Chambers*

ELEVATION



h *Cast Iron Sulphur Pan*
i *Cast Iron Nitre Dishes*

SEGMENT



GROUND PLAN

WILLIAM MACKENZIE, LONDON, EDINBURGH & GLASGOW.

APPARATAS FOR THE MANUFACTURE OF CHLORINE BY WELDON'S PROCESS.

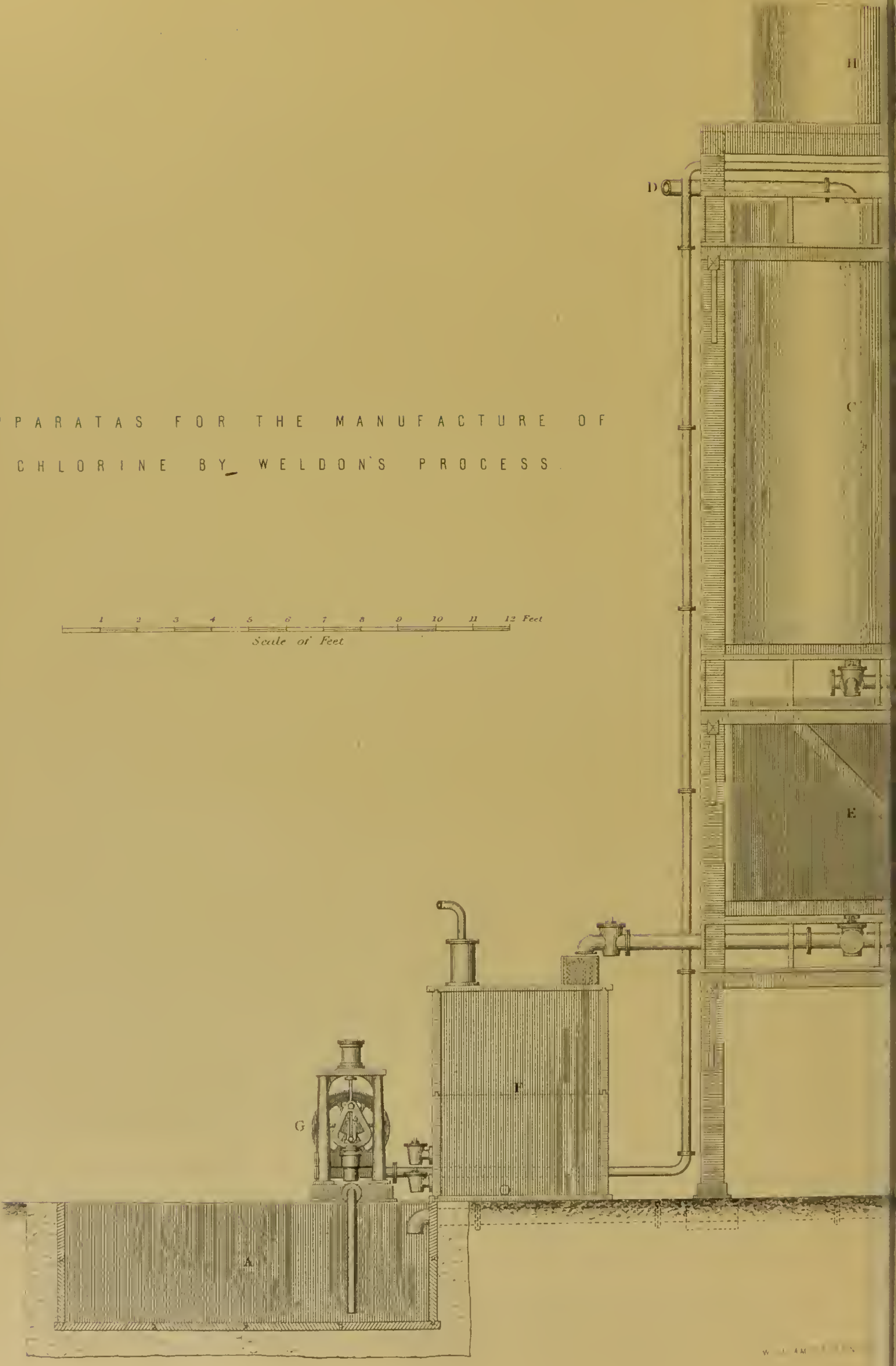


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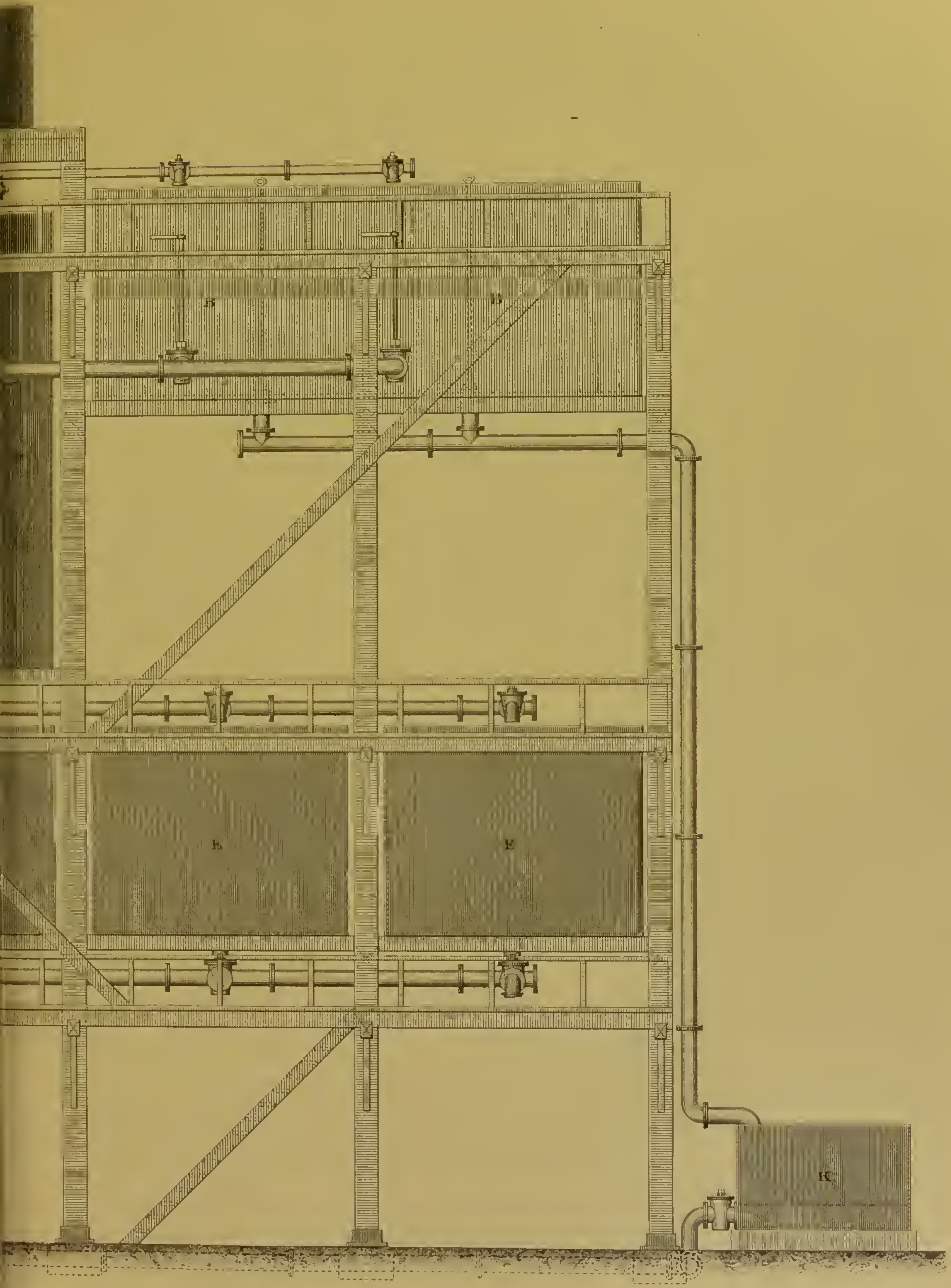
PLATE I.



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SECTION OF A WHISKY DIST

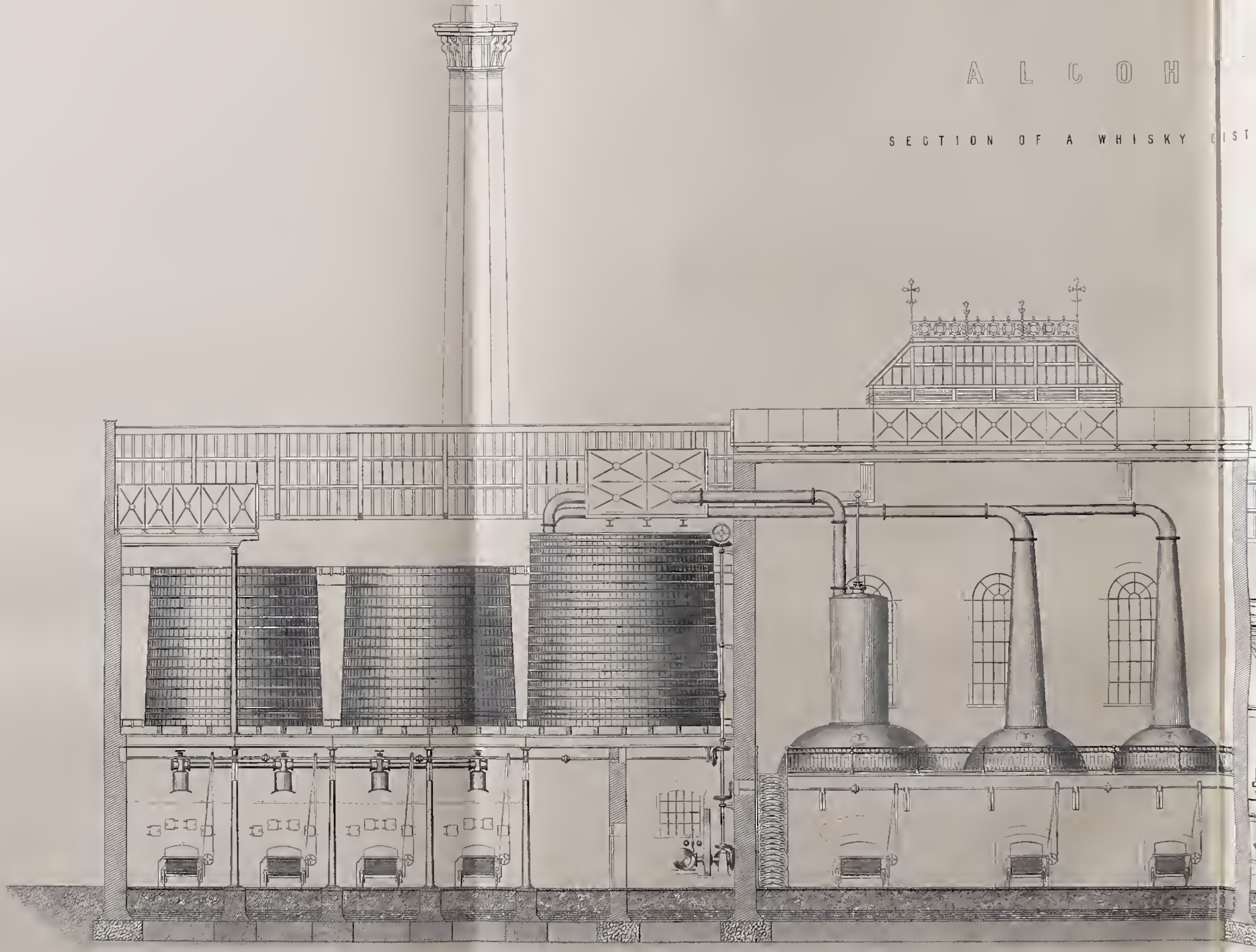

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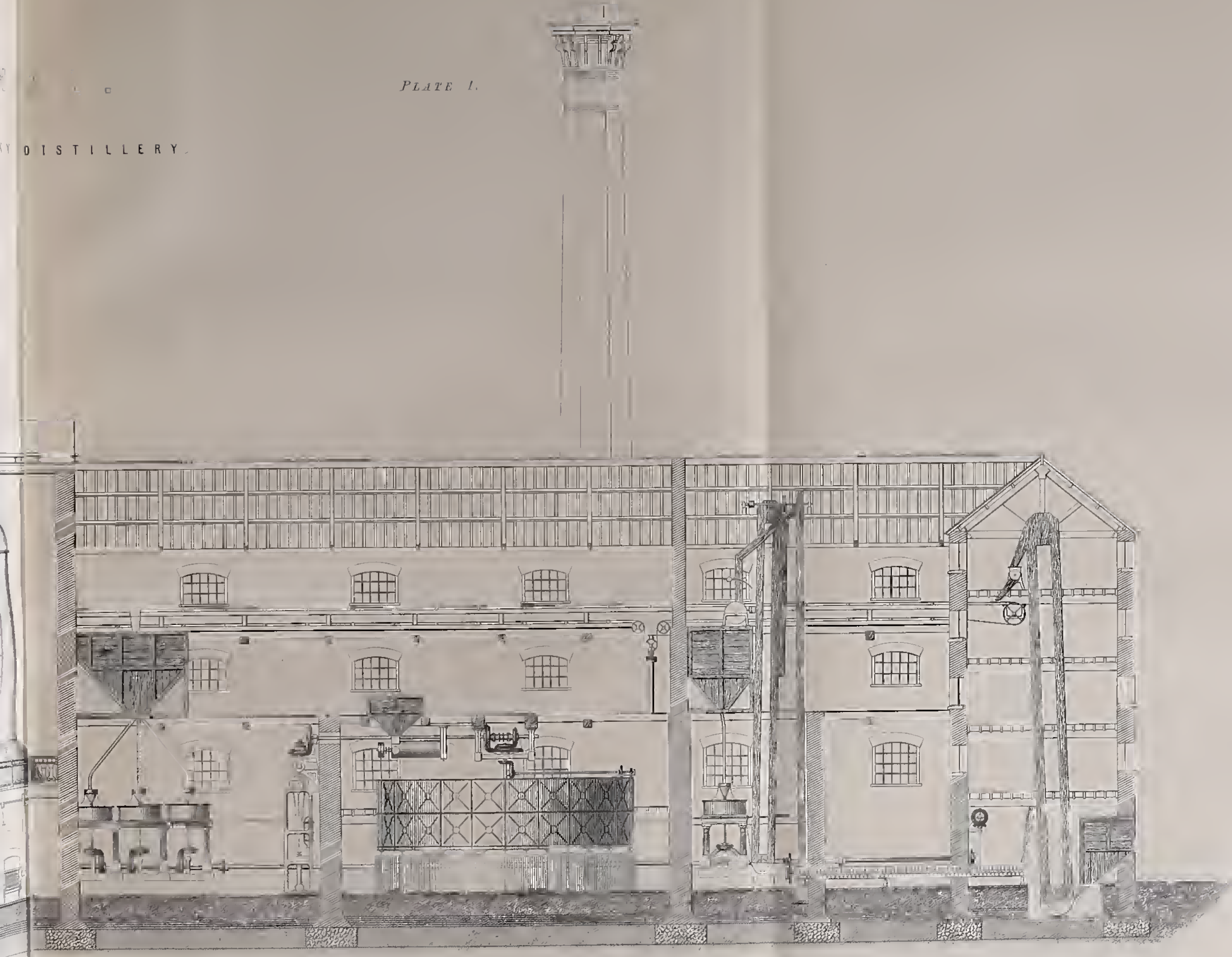
PLATE I.

KY DISTILLERY.



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CHEMISTRY,

THEORETICAL, PRACTICAL, AND ANALYTICAL,

AS APPLIED TO THE

ARTS AND MANUFACTURES.

ACETIC ACID.

ACETIC ACID. — *Acide Acétique*, French; *Essigsaure*, German; *Acidum Aceticum*, Latin: *Eisel*, Saxon; $C_2H_4O_2 = \left.\begin{matrix}C_2H_3O\\H\end{matrix}\right\} O$, or $C_2H_3O_2H$. The hydrate or hydrated oxide of the as yet unisolated radicle acetyl (C_2H_3O). Acetic acid was formerly supposed to be a trioxide of the radicle C_2H_3 (old notation C_4H_3), the hydrated acid being a compound of this oxide ($C_4H_3O_3$) with water ($C_4H_3O_3,C_2H_3HO$). The assumption of the radicle acetyl enables the reactions of acetic acid to be represented very simply. Thus when potassium carbonate is added to dilute acetic acid, the basic atom of hydrogen is replaced by the metal, and potassium acetate is formed — $\left.\begin{matrix}C_2H_3\\K\end{matrix}\right\} O = KC_2H_3O_2$. In fact, acetic acid may be viewed as a molecule of water in which half the hydrogen is replaced by acetyl, and when the remainder of this hydrogen is replaced by a metal an acetate is produced.

Ordinary vinegar is dilute acetic acid, contaminated with various vegetable impurities. In this form it has been known from the earliest times. Moses mentions it (Numbers vi. 3). Hippocrates made use of it as a medicine; and though until very recently there was no definite knowledge as to the cause of its production and the mode of its formation, there can be no doubt that it was in very general use, and that most of its properties as regards its action on metals, &c., had been investigated at a very remote period.

The alchemists were acquainted with this acid in a concentrated state, and obtained it by distilling copper acetate (verdigris). Both Geber and Stahl describe this process. The product of the further rectification of the liquid was termed *Radical vinegar*, *Spiritus Veneris*, *Venus' vinegar*, *Spiritus aeruginis*, &c.

Acetic acid was first obtained as a pure hydrate by Löwitz in 1793. Subsequently it was observed by Dr. J. Davy that spongy platinum, in contact with the vapour of alcohol, became incandescent, and generated this acid. Döbereiner further studied the nature of the acid, and proved that the alcohol was oxidized at the expense of the atmospheric air, producing acetic acid and water, and that no carbonic acid was formed—thus pointing out the fallacy of the opinion held by the chemists of his time, that carbonic acid was one of the products of the acetous fermentation. Further, he showed that, for the complete oxidation of one atom of alcohol, four atoms of oxygen were required.

Glacial Acetic Acid.—Pure acetic acid ($C_2H_4O_2$) is at ordinary temperatures a crystalline solid. It is obtained by distilling finely powdered anhydrous metallic acetates with an equivalent quantity of concentrated sulphuric acid or potassium bisulphate. Thus with acetate of potassium and sulphuric acid:—

$$2KC_2H_3O_3 + H_2SO_4 = 2C_2H_4O_2 + K_2SO_4,$$

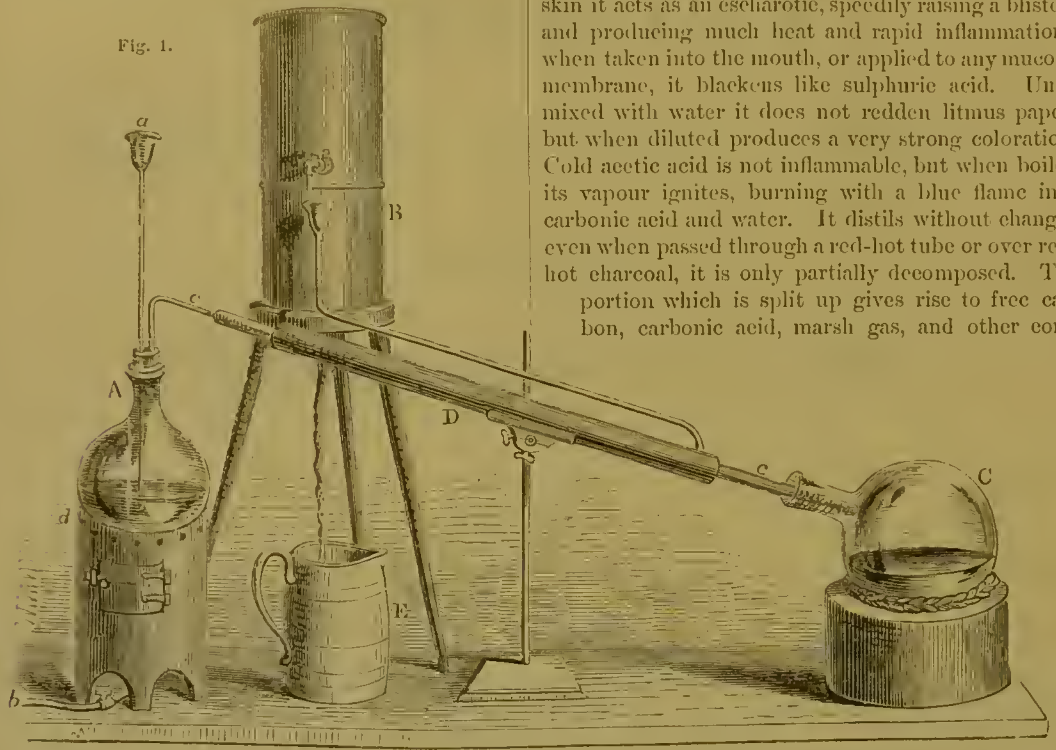
or with potassium bisulphate:—

$$KC_2H_3O_2 + KHSO_4 = C_2H_4O_2 + K_2SO_4.$$

The proportions are 98 of potassium acetate, 82 sodium acetate, 79 calcium acetate, or 163 lead acetate, to 49 of sulphuric acid (H_2SO_4), or 136 of potassium bisulphate ($KHSO_4$).

Fig. 1 is an apparatus well adapted for this purpose. A is a flask, closed by a cork, which receives two tubes; one of these is a funnel tube, *a*, through which the liquid bodies are introduced, and the other conducts the vapours to the condenser. This consists of an outer case, D, through which the tube, *c c*, connected with the bent tube from the flask, A, passes. The condenser is cooled by keeping a stream of water running constantly from the reservoir, B; C is a flask to receive the condensed vapours; *d*, the gas lamp which heats the liquid in A; *b* the gas pipe, and E the vessel into which the heated water flows from the condenser.

Fig. 1.



The odour of pure acetic acid is peculiarly suffocating, but when mixed with air it is very agreeable. It is a powerful restorative when applied to the nostrils in impending fainting. It is nearly as acrimonious as sulphuric acid; when dropped on the skin it acts as an escharotic, speedily raising a blister, and producing much heat and rapid inflammation; when taken into the mouth, or applied to any mucous membrane, it blackens like sulphuric acid. Until mixed with water it does not redden litmus paper, but when diluted produces a very strong coloration. Cold acetic acid is not inflammable, but when boiled its vapour ignites, burning with a blue flame into carbonic acid and water. It distils without change; even when passed through a red-hot tube or over red-hot charcoal, it is only partially decomposed. The portion which is split up gives rise to free carbon, carbonic acid, marsh gas, and other combustible gases, acetone, naphthaline, hydrate of phenyl, and benzol.

The specific gravity of acetic acid is 1·0635 at 15° C. (Mohr). It boils at 117° C. to 119° C., solidifies at 15° C., and melts at 16° C. On first addition of water heat is evolved, and a contraction of volume ensues until (20 to 22 per cent. of water having been added) a hydrate is formed, having the composition $C_2H_4O_2,H_2O$. This acid has the sp. gr. 1·0748, and boils at 104° C. On adding more water the density of the liquid again diminishes. Hence in determining the strength of acetic acid, the density is no criterion of the amount of acetic acid present.

The following table, drawn up by Mohr, shows that the specific gravity test answers very well, when it is required to determine the amount of anhydrous acid in dilute solutions; but when the acid increases in strength it is very fallacious:—

Per Cent.	Sp. Gr.	Per Cent.	Sp. Gr.	Per Cent.	Sp. Gr.	Per Cent.	Sp. Gr.	Per Cent.	Sp. Gr.
100	1·0635	80	1·0735	60	1·067	40	1·051	20	1·027
99	1·0655	79	1·0735	59	1·066	39	1·050	19	1·026
98	1·0670	78	1·0732	58	1·066	38	1·049	18	1·025
97	1·0680	77	1·0732	57	1·065	37	1·048	17	1·024
96	1·0690	76	1·0730	56	1·064	36	1·047	16	1·023
95	1·0700	75	1·0720	55	1·064	35	1·046	15	1·022
94	1·0706	74	1·0720	54	1·063	34	1·045	14	1·020
93	1·0708	73	1·0720	53	1·063	33	1·044	13	1·018
92	1·0716	72	1·0710	52	1·062	32	1·042	12	1·017
91	1·0721	71	1·0710	51	1·061	31	1·041	11	1·016
90	1·0730	70	1·0700	50	1·060	30	1·040	10	1·015
89	1·0730	69	1·0700	49	1·059	29	1·039	9	1·013
88	1·0730	68	1·0700	48	1·058	28	1·038	8	1·012
87	1·0730	67	1·0690	47	1·056	27	1·036	7	1·010
86	1·0730	66	1·0690	46	1·055	26	1·035	6	1·008
85	1·0730	65	1·0680	45	1·055	25	1·034	5	1·007
84	1·0730	64	1·0680	44	1·054	24	1·033	4	1·005
83	1·0730	63	1·0680	43	1·053	23	1·032	3	1·004
82	1·0730	62	1·0670	42	1·052	22	1·031	2	1·002
81	1·0732	61	1·0670	41	1·051	21	1·029	1	1·001

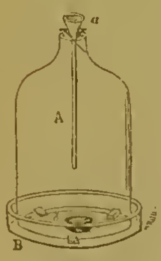
Acetic acid mixes with water in all proportions, and dissolves many essential oils, hydrocarbons, such as camphor, guiacum, and various resins, fibrinic of blood, gelatine, &c., and many other bodies which are insoluble in water. It is this peculiar power of solution which renders it when properly diluted and moderately used so valuable as a condiment. Free acetic acid is always in the stomach when in a healthy state, and the substitution for it of lactic acid is one of the most common accompaniments of indigestion. In cases of poisoning by alcohol, or with narcotic poisons, the power of acetic acid over the nervous system is very great. In the latter case care must be taken that the narcotic is first removed, as otherwise a powerful acetate would be formed; but after removal of the poison few substances more successfully combat the secondary symptoms.

In all its states the metal platinum possesses in a very high degree the property of condensing gases upon its surface. A clean plate of platinum condenses oxygen and hydrogen upon itself with such force, that if placed in a mixture of combining volumes of the two gases sufficient heat is disengaged to fire the remnant of the gases. When very finely divided platinum is used, the surface is so greatly extended that the phenomenon becomes still more striking from the rapidity with which the action is effected.

Spongy platinum is obtained by heating to redness the double chloride of platinum and ammonium. From its minute state of division it condenses within itself several hundred times its volume of oxygen; consequently, when the vapour of alcohol comes in contact with this body, a supply of oxygen in a concentrated state is presented to it, and the platinum, without losing any of its own inherent properties, effects chemical combination, the alcohol undergoing slow combustion, and being converted into acetic acid. In order that the reaction may continue, it is of course necessary to present fresh oxygen to the platinum, to replace that which is withdrawn. The two actions then go on side by side.

This can be illustrated by an apparatus similar to Fig. 2. A is a bell glass, through the mouth of which a long funnel, *a*, passes; the lower end of this funnel terminates in a fine point, so that the alcohol poured in may percolate very slowly. The vessel is placed upon supports, *b*. within a dish, B, in which is a saucer or small flat basin, containing the spongy platinum. The interstice between the bottom of the dish, B, and the bell, A, serves for the circulation of air in the jar. On pouring the alcohol through *a*, in the course of a short time the odour of acetic acid is perceived at the mouth, from the acetic acid vapours which are generated. These condense on the sides of the jar, and trickle to the bottom, where they collect in B. It is advantageous for the success of the experiment to have the alcohol heated to about 32° C. (90° Fahr.) when it is poured in. In Germany and other continental countries, where the duty on alcoholic liquors is not so high as in England, this method was formerly carried on as a commercial process, but it was found that so much of the alcohol was converted into aldehyde and lost by volatilization, that the manufacture was ultimately abandoned. The reaction is conceived to take place by two steps—

Fig. 2.

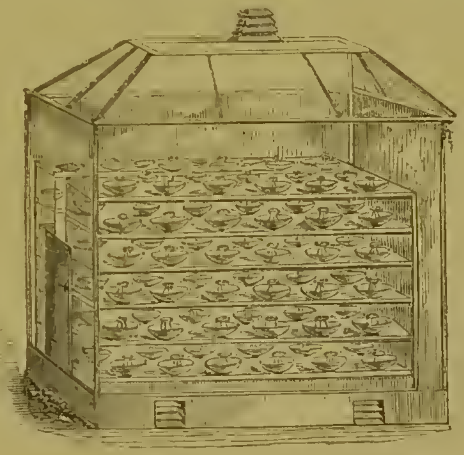


$$\overset{\text{Alcohol.}}{C_2H_6O} + O = \overset{\text{Aldehyde.}}{C_2H_4O} + \overset{\text{Water.}}{H_2O}$$

$$\text{and } \overset{\text{Aldehyde.}}{C_2H_4O} + O = \overset{\text{Acetic Acid.}}{C_2H_4O_2}.$$

On the large scale, a glass case, or one of wood, the roof of which is of glass to admit the heat of the sun, is constructed. In the interior of this case the shelves are arranged, twelve inches apart, on which a series of shallow glazed earthenware or porcelain dishes are placed; in each is a porcelain or stoneware tripod, bearing a watch-glass or small dish, containing the spongy metal. The alcohol is poured into these vessels, and no more than an inch and a half, or two inches, should intervene between the platinum in the watch-glasses and the surface of the alcohol in the flat dishes. If there be no arrangement to supply an influx of air in the place of that which becomes deoxidized, no more alcohol should be operated upon than the volume of air in the apparatus is capable of converting into acetic acid. This quantity may be inferred from the fact that 110 grains of alcohol require for their complete oxidation 1000 cubic inches of air, producing 120 grains of anhydrous acetic acid, and about 65 grains of water. If a draught be instituted, by which the vitiated or nitrogenous portion of the deoxidized air is withdrawn, and fresh quantities supplied by means of air-passages in the lower part of the chamber, the necessity of observing, with the accuracy previously mentioned, the amount of alcohol submitted to oxidation, is obviated. This arrangement is shown in Fig. 3. The temperature of the air in the case is raised to about 32° C. (90° Fahr.) by means of steam pipes or flues from a fire adjacent to the apparatus, similar to those in vineries in this country. Oxidation of the alcohol then commences, which is ascertained by the pungent odour of the acid. The

Fig. 3.



elevated temperature converts a portion of the alcohol into vapour, which, on coming into contact with the moistened platinum, undergoes incipient combustion, giving rise to the acid. These vapours condense, and are collected, for the most part, in the dishes; the remaining quantity trickles down into a receiver at the bottom of the case. In this manner the whole of the alcohol, in a comparatively short time, is converted into acetic acid; and so long as a supply of fresh air is kept up in the chamber, the spongy platinum continues to oxidize the liquid.

With a case of 12 cubic feet capacity, and 7 or 8 oz. of platinum black, 1 lb. of absolute alcohol may be acetified daily; and with a provision of 24 or 30 lbs. of spongy platinum, and a proportionate sized case, 300 lbs. of alcohol may be oxidized in the same time, producing an acid of the purest kind.

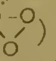
In every instance where alcohol or fermented alcoholic liquors are acetified, the principle of the conversion is the combustion of the alcohol of those liquors by combination with oxygen. This result is also attained when alcoholic liquids are exposed to the air at a slightly elevated temperature, in contact with a body in the state of fermentation, and in several other ways—which change has been called the *acetous fermentation.* In this manner wine, brandy, beer, and in fact all liquids which undergo the vinous fermentation, are converted into solutions containing acetic acid; and many liquids apparently pass at once into the acetous fermentation, especially those eviscerating a quantity of mucilage and very little sugar. Alcohol in a pure state does not suffer the acetous fermentation, but if it contains vegetable matter a metamorphosis occurs on its exposure to the air; hence the cause of the souring of wines, and the reason why weak ones do so sooner than strong, the former containing little spirit and much vegetable matter, while with the latter the reverse is the case.

In all cases of acetous fermentation, where a quantity of liquid is exposed to the air, oxidation takes place at the surface only, and this occasions the conversion of their alcohol into acetic acid to extend over several weeks, or even months. Heat very much accelerates the change, inasmuch as a portion of the alcohol is converted into vapour, and this, carrying with it some of the ferment in a state of eremacausis, communicates the same property to the vapour likewise, and acetic acid results; besides, imperceptible currents form in the liquids, by which fresh surfaces are always exposed till the work is completed. Spirituous liquors, on being exposed to the air in a state of fermentation, or with a ferment added to them, however clear they may be at first, speedily become turbid, and slimy filaments appear through the solutions, which gradually adhere and rise as a spume to the surface. When this spume incrassates, it precipitates to the bottom of the vessel, and is called *mother of vinegar.* During the formation of this body an elevation of temperature is observed, a peculiar aromatic odour is evolved, and an acid reaction acquired; towards the end of the operation the temperature falls to about that of the surrounding air, the liquor clarifies, and when it is siphoned off constitutes the well-known liquid, Vinegar.

It was formerly imagined that the formation of vinegar was accomplished by a peculiar fermentation, which was called *acetous*, in contradistinction to *lactic*, *vinous*, *butyric*, *mucic*, &c. It is now held by most chemists that, although in certain processes for the manufacture of vinegar fermentation takes place, yet this is rather the accompaniment than the cause of the chemical change.

At the close of the operation the nitrogenous organic matters contained in the liquor, and which have produced fermentation, are found at the bottom of the vat as a white, gelatinous, fungoid plant, to which the name of *Mycoderma vini* was given by Mulder. The experiments of Pasteur conclusively show that the presence of this fungus is essential to the formation of vinegar from alcohol solutions by the fermentation processes. It is also absolutely necessary that the spores should remain at the surface of the liquor. The plant may be sown by adding to the liquor a portion of a solution already containing the fungus. Where spontaneous acetification sets in it is because germs have fallen into the solutions from the air, where these and many others are almost always floating. The plant derives its nourishment from the albuminous matter and mineral salts contained in the mother liquor. Pure aqueous alcohol does not undergo acetification, however long exposed to the action of ordinary atmospheric air, because any germs which it acquires die for want of sustenance. When, however, Pasteur supplied the necessary nutriment by adding small quantities of ammonium phosphate (to furnish nitrogen) and alkaline and earthy phosphates, the atmospheric germs became fertile, and the alcoholic was converted into acetic solution. Fermentation also removes the too readily decomposable organic bodies, and is therefore essential to the production of saleable vinegar; but the amount of acidification depends entirely on the amount of alcohol which has been oxidized into acetic acid, and this oxidation appears to be entirely independent of those changes which merely result in the clarification of the liquor.

Buchen has shown that no *Mycoderma aceti* (*Mycoderma vini*) was to be found in the contents of a vat for oxidation of alcohol by air which had been in use for twenty-five years. The experiments of John Davy and Döbereiner tend to the same conclusion. The vinegar plant and allied vegetations probably act like spongy platinum and platinum black, rendering the oxygen more active by condensation in their pores in the course of their growth, and bringing it in an active state (?ozone) in intimate contact with the molecules of alcohol.

This view is still further borne out by the fact that ozone (probably triatomic oxygen, ) converts alcohol into acetic acid with very great rapidity. In 1869 Widemann erected a factory at Boston, U.S., to carry out this reaction commercially. His first idea was that the fusel oil in whiskey might be greatly reduced in amount by passing over it a stream of ozonized air. The results he obtained were surprising, and by the adoption of proper mechanical contrivances he so decomposed the whole of the fusel oil in maize whiskey, that in eight minutes the latter was, in the opinion of experts, equal to a spirit six years old. In 1870, 300 barrels of 40 gallons each were thus treated weekly, and the works were being extended.

He was naturally led to experiment upon diluted alcohols, and found that their oxidation was rapidly effected by ozone. He says:—"By adding water to maize whiskey and treating it as before, in almost as short a time (eight minutes) I obtained its complete conversion into vinegar." The best result was obtained by operating on a mixture of U.S. proof whiskey with seven times its weight of water.

On the 20th of April, 1871, works were opened at White Plains to manufacture vinegar by this means, which turned out from their commencement thirty barrels of vinegar daily, in a fit state to be immediately employed for pickling. In January, 1872, the same works were producing daily ninety barrels of vinegar of 40 gallons each.

Lavoisier's theory, that acetic acid is alcohol plus oxygen, and that the change effected by the so-called acetous fermentation is the oxidation of the alcohol, is now universally accepted by chemists.

Of all the volatile organic acids, acetic acid is the only one having the property of dissolving lead monoxide (PbO) and copper monoxide, forming basic acetates of these metals. The dilute acetic acid, or distilled vinegar used in pharmacy, should therefore always be examined for copper and lead, these impurities being apt to be contracted from the metallic vessels sometimes employed in the process.

Acetic acid exhibits the reactions of the fatty acids. It is not itself decomposed by electrolysis, being almost a non-conductor of electricity. A solution of potassium acetate is, however, split up by the voltaic current, with formation of dimethyl (ethane) and potassium carbonate:—

$$2KC_2H_3O_2 + H_2O = C_2H_6 + H_2 + CO_2 + K_2CO_3.$$

When exposed to a gradually rising temperature the vapour of acetic acid dilates abnormally, as may be seen from the following observations of Bineau:—

Degrees C.	Density.	Degrees C.	Density.
21	3·95	200	2·22
125	3·2	230	2·09
160	2·48	238	2·08

At 230° C. and upwards 1 atom of acid yields 2 volumes of vapour in accordance with the usual law. Beyond 238° the vapour undergoes no change of volume until the acid itself is decomposed.

Chlorine attacks acetic acid very slowly in diffuse light; in the sunlight its action is more rapid, chloracetic (C_2H_3ClO, Hofmann) and trichloracetic ($C_2HCl_3O_2$, Dumas) acids being formed. In like manner a mixture of bromine and acetic acid produces dibromacetic acid ($C_2H_2Br_2O_2$) in sunlight. Heated with bromine in a sealed tube, bromacetic acid ($C_2H_3BrO_2$) is formed, Perkin and Duppa.

Acetic acid is unaffected by nitric acid. Heated with sulphuric acid it is blackened, and carbonic acid and sulphurous acid are disengaged. Sulphuric anhydride dissolves in acetic acid, and if the mixture be kept for a few days at a temperature between 60° and 75° C. sulphacetic acid ($C_2H_4SO_5$) is produced.

When ferric chloride (sesquichloride of iron, Fe_2Cl_6) is added to acetic acid, and the acid then nearly saturated with ammonia, or if a neutral acetate is mixed with ferric chloride, the fluid acquires a deep red colour, owing to the formation of ferric acetate. Mercurous nitrate (nitrate of suboxide of mercury) precipitates mercurous acetate in white scales, which, on being heated, is partially resolved into metallic mercury. In presence of an excess of potassa, auric chloride (trichloride of gold, $AuCl_3$) is reduced on treating with acetic acid, and metallic gold deposited.

Acetic acid has been formed synthetically by Wanklyn and also by Berthelot. Wanklyn prepared sodium methyl ($NaCH_3$) by the action of sodium on a solution of zinc methyl in ether; he then passed into it a current of carbonic acid, when sodium acetate was formed:—

$$CO_2 + NaCH_3 = NaC_2H_3O_2.$$

Berthelot formed potassium acetate by heating acetylene dichloride ($C_2H_2Cl_2$) with aqueous potash to 230° C., or with alcoholic potash to 100° C. for ten hours:—

$$C_2H_2Cl_2 + 3KHO = KC_2H_3O_2 + 2KCl + H_2O.$$

The strength of glacial acetic acid is commonly estimated volumetrically by a standard solution of caustic soda. The change in the colour of the litmus not being very strongly marked, Rüdorff has proposed a new method, which he states to be very accurate, and which consists in determining the solidifying point of the acid.

WINE VINEGAR.—*Weinessig*, German; *Vinaigre*, French.—This species of vinegar is chiefly fabricated in wine-growing localities, or where grapes are abundant, from wines which have become acid, full flavoured wines giving the best table vinegar, the principal factories being at Orleans in France. The building where the work is carried on is called a *vinaigrerie*, and has always a southern aspect. The casks, called *mothers*, which are employed, hold from fifty to one hundred gallons, and rest upon strong wooden frames, supported by pillars of wood or stone of eighteen inches in height. Several such casks are placed in rows, and when acetification is carried on in the open air, eight, ten, fifteen, or twenty such ranks constitute what is termed a *vinegar-field*. Two holes are bored in the upper surface of the front end of each cask; the larger serves to charge the cask with wine, as also to draw off the vinegar when formed, and the smaller allows an influx or efflux of air as the cask is emptied or charged. The chief aim of the person who wishes to carry on a vinegar factory is to have a good fermenting-room, where the wines are exposed to an even temperature, having a copious supply of atmospheric air and free ventilation, in order to replace as rapidly as possible the air which has been exhausted of oxygen, and to carry off the carbonic acid set free, the air-holes for this purpose being constructed so as to admit of being closed in windy weather, or when the temperature of the room is depressed. The walls of the apartments are of brick, or some non-conducting material, and lined with lath and plaster.

Low-roofed apartments are the most suitable. When there is a high ceiling it is necessary to elevate the mothers, in order that they may occupy the higher strata of warm air. This trouble is dispensed with when the roofs are low. Experience has pointed out that in high-roofed apartments, where the tuns are placed at different levels, the uppermost work off quicker and better than the others. In the event of new mothers or vessels being used, it is needful to fill them one-third full with the strongest vinegar at a boiling temperature. This forms the stock, or true *mother*. The charges of wine added each time are two and a half gallons to every cask, and an interval of eight days is allowed for the acetification of each charge, before adding another of fresh wine. This treatment is continued—of charging and allowing eight days to work it off—till the casks are more than half full. One-third of the contents of each mother is then siphoned off—in some factories only 10 gallons—and run into the store tuns, and the process repeated anew of charging every eight days till the mothers are refilled, as before. Some manufacturers do not suffer the vinegar to remain in the mothers till they are two-thirds full, but siphon off at the end of every sixth or eighth charge 12 or 15 gallons of vinegar. The mothers should never be charged with more than the above quantity, in order to carry on a steady and efficient mode of acetification. Occasionally it happens that eight days are not sufficient to finish every charge. This is more unaccountable from the fact that the backward casks receive the same amount of care, and have the same temperature, as those which work well. It often occurs that the casks in the warmest part of the room are those which are backward, or *lazy*, as they are termed. In this event nothing remains but to empty such mothers of their contents and fill them with hot strong vinegar, when, on adding fresh charges, the acetous fermentation recommences and goes on as briskly as in the rest. Sometimes fresh quantities of a stronger wine and an increase of temperature are supplied, to quicken the fermentation in such casks, which mode is often successful. The *laziness* of the mothers is attributed to very vague and unsatisfactory causes, some regarding it as the effect of the electrical state of the casks and liquid.

It has been recommended to isolate the mothers as much as possible, and to use little or no iron in the construction of the casks. To ascertain if the liquor has fermented, the following experiment is resorted to:—A white rod, bent at one end, is plunged into the mothers and drawn out in a horizontal direction; if the rod be covered with a thick white froth—flowers of vinegar—the operation is said to be terminated; if the froth be reddish-brown, more wine is added, and the temperature increased till the whole is acetified. In summer the natural heat is sufficient, but in winter the mothers are heated by means of a stove to about 80° Fahr. (27° C.). The prevailing temperature should range between 75° and 80° Fahr. (24° and 27° C.). When proper attention has been made to the manufacture, the mothers usually work off double their contents of vinegar annually. The precipitation of the insoluble matters of ferment, the accumulation of *mother of vinegar*, and the deposit of tartar from the wine, fill the casks to such an extent that it is indispensable to empty the whole of them, and free them from these deposits every six or eight years; and often the entire factory needs renovation, but good casks will last for a period of twenty-five years.

The wine, if it be ropy, is introduced into a large tun filled with beechwood shavings, through a funnel opening in the cover, and allowed to repose for some time, whence it is afterwards drawn off by a tap in the lower part of the tun, and supplied to the mothers as required. Frequently, when weak wines are employed, from the proportionably large amount of vegetable matter they contain, it happens that the resulting vinegar is ropy and turbid; in these instances it is necessary to pass it through the clarifying or *fining* tun, and the advantage of having an average vinegar is gained.

The old method, introduced by Boerhaave, is still practised in Holland, in France, and on the Rhine. Two large tuns or vats, about 9 feet high and 4 feet in diameter, are supported on stands about 12 inches from the floor. Within 1 foot from the bottom of each vat is a perforated bottom, or wooden grate, resembling that of a riddle; on this a quantity of fresh cuttings from the vine, willow twigs, &c., is placed, and pressed closely together, the remainder of the vats being filled with *rapes*—the footstalks of the grapes—and light vine branches. Both vats are left open for the admission of the external air. They are then charged with wine; one is completely filled, the other only half. The two are left at rest for twenty-four hours at 75° Fahr. (24° C.), after which the half-filled vat is replenished from that already full, till the latter contains only half its contents of liquor. Twenty-four hours elapse before the liquid is transferred from the filled to the half-filled vat.

The process of transferring the liquid into the vats alternately is repeated every twenty-four hours until the vinegar is made. Towards the third or fourth day an internal effervescence is observed in the half-filled vat, which is accompanied by a sensible elevation of temperature, increasing gradually each successive day. On the other hand, the temperature and fermenting action of the filled vat are but sluggishly progressing, so that the intestine motion takes place only on alternate days in each vat. The completion of the process is known by the decreased temperature and abated action even in the half-filled vat. The vinegar is then drawn off into casks, and left in a cool situation till it clarifies. During summer the time occupied is fifteen days, but in winter the acetification extends over a longer period. The temperature of the half-empty vat should never exceed 80° Fahr. (27° C.); if it rises to 84° Fahr. (29° C.) the liquor is to be transferred every twelve hours, and an oaken cover placed on the half-filled vat in order to check the fermentation, otherwise the aldehyde, or half-made vinegar, will be dissipated, and only a vapid fluid remains, sour, but effete. If the whole be kept at 83° Fahr. (28° C.), and the menstruum transferred every twelve hours, the acetification will be effected in eight or ten days. According to DUMAS, the best French vinegar is made of good wine, which is put into a cask already containing vinegar, and to which atmospheric air has constant access. As acetification proceeds more wine is added at intervals, and when the whole has become vinegar it is drawn off to the amount of the wine used, and the process is repeated. Its strength, flavour, and colour depend upon the characters of the wine employed. The temperature of the factory is maintained at 86° Fahr. (30° C.). As the acetification becomes more complete the wine loses its peculiar odour and flavour, and acquires the sweet sharpness of vinegar. The weight of the liquor also increases. The absorption of oxygen, and its consequent conversion from the gaseous to the liquid state, causes a rise in temperature proportionate to the rapidity of the process.

MALT VINEGAR.—*Malz-Getreide* or *Bieressig*, German.—In this country the chief part of the vinegar is made from *malt wash*, or *gyle*, prepared by operating upon the materials in the annexed proportions:—Six bushels of good barley malt, properly ground, are mashed with 40 gallons of water at 160° Fahr. (71° C.), permitted to repose till the solid matter settles down, the solution drawn of, and a fresh quantity of water, say 40 gallons, at 180°, added to the residue, well agitated for a short time, allowed to settle, and then siphoned off as before. To take up all the soluble matters, the third washings may be performed with boiling water. On the whole, not more than 100 gallons of wash is to be used in extracting the soluble matters. When the solution has cooled to about 75° Fahr. (24° C.), it is well agitated with 4 gallons of yeast of beer; and after thirty-six to forty hours racked off into casks, and placed in the vinegar stoves or apartments, the temperature of which should range from 70° to 77° Fahr. (21° to 25° C.). The casks should be placed on their sides, the bungholes opened, and a circulation of air kept up in each cask by means of an orifice bored at each end of the cask, near its upper edge. Since the temperature of the liquid is somewhat less than the surrounding atmosphere, in consequence of the evaporation at the surface, an efflux of cold air takes place at the holes while the warm air enters at the bung, and thus a constant current is kept up.

This manufacture is frequently effected by *fielding*. In this case, as the term implies, the process is conducted in the open air. The casks rest on strong frames, one foot and a half high, being supported by firm pillars of brickwork or wood. Six or eight rows of these are arranged parallel to each other, with a narrow walk between each pair of rows; a sluice is placed along the casks into which the vinegar is siphoned, whence it flows into the store tuns in the magazine; and a flexible tube, or hose, supplies the *wash* from the great tun in the brewhouse. The bungholes are left open in dry, and are loosely covered with a tile in rainy weather. One-third of each *mother* is left empty for the circulation of air, so as to oxidize the alcohol as it generates in the wort. Three months are required to complete the process, and render the vinegar marketable.

The process of malting materially changes the character of the grain. The barley is steeped in water until it begins to swell, after which it is piled up in heaps (technically *couched*). Germination sets in, oxygen is absorbed from the air, producing a kind of slow combustion, accompanied by its necessary elevation of temperature. When this has proceeded far enough, further growth of the barley is prevented by heating the sprouted grain in suitable kilns. During germination a peculiar nitrogenous substance, termed diastase, is produced, which reacts on the starch of the grain, converts a portion of it into grape sugar (glucose, $C_6H_{12}O_6$), and renders it soluble, the starch taking up the elements of water:—

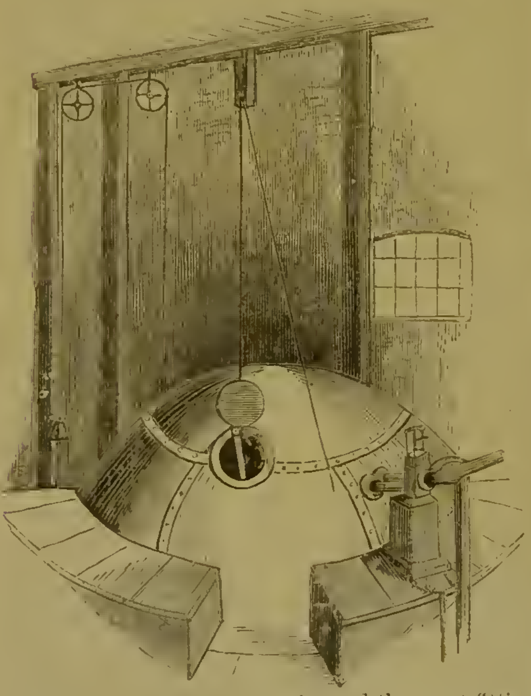
$$\overset{\text{Starch.}}{3C_6H_{10}O_5} + H_2O = \overset{\text{Glucose.}}{C_6H_{12}O_6} + \overset{\text{Dextrin.}}{2C_6H_{10}O_6}.$$

This conversion into sugar, called the *saccharine fermentation*, is the most important step in the preparation of beer, whiskey, and malt vinegar; for it is from this sugar that vinous fermentation produces alcohol, the parent of vinegar. Hence the early processes in an ale brewery, a malt distillery, and a malt vinegar works, are similar.

In the factory about to be described the malt is hauled up out of the waggons into the upper floors of the brewhouse. Here openings, placed in different directions, permit of the malt being poured down into the large bins, whence it is removed when a brewing is to commence. Vinegar-makers and distillers, as well as beer-brewers, give the name of *brewing* to the extraction of a saccharine liquor from malt. The quantity required for one brewing being measured out, and taken from the bins in sacks, it is poured through *hoppers*, or funnels, at the top of the grinding apparatus, by which the malt is reduced to meal. The apparatus consists of millstones and crushing-rollers, either or both of which can be employed as may be deemed advisable. In the one case a flat circular stone rotates and crushes beneath it the malt, which flows between it and a lower fixed stone: in the other the malt, after flowing through a shoot or trunk from the hopper, falls on a wire grating, where it becomes depurated. It then passes between two cast-iron rollers, rotating nearly in contact, by which means it is crushed into fragments. Various ingenious contrivances are adopted for stopping the circular motion of the rollers if any hard substance gets between them.

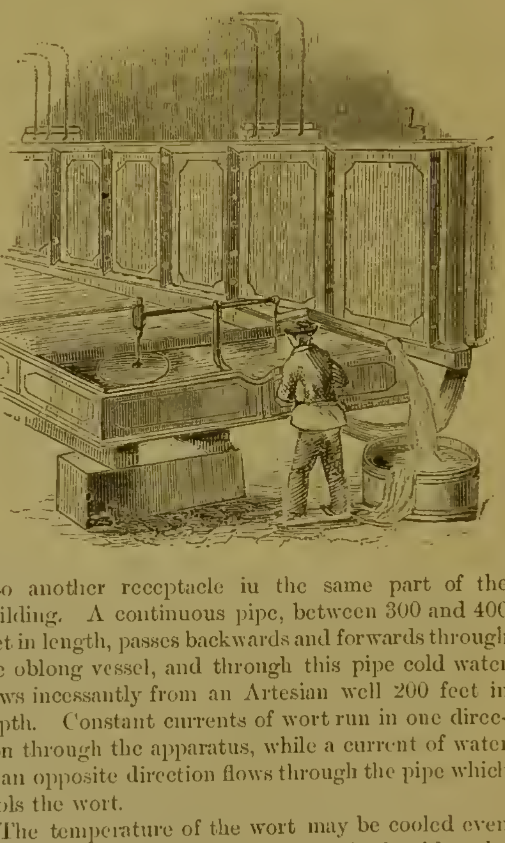
When the malt is crushed or ground, it falls through a hose or trunk into the mash-tuns in the floor beneath. These mash-tuns are similar to those used at large breweries and distilleries, but smaller in size. They are circular vessels, with a central *stirrer*, or instrument for keeping in constant agitation the ingredients contained in the tuns—the stirrer being worked by a steam-engine. It is in these vessels that the *saccharine fermentation* proceeds. The diastase, which is very soluble, acts upon the unaltered starch of the grain, and converts it into dextrin and grape sugar, as before mentioned. This grape sugar (sometimes also called glucose, dextrose, and maltose) is the sweet principle which subsequently yields to the brewer his beer, to the distiller his spirit, and to the vinegar-maker his acetic acid; and it may well be supposed that every precaution is taken, and every investigation made, as to the extraction of the greatest quantity and the most fitting quality of this important agent. The quantity of water required for a given quantity of malt, and the temperature at which the water is used, vary in each particular branch of manufacture, according to the strength of the *wort* required. The arrangements for settling these are very exact and ingenious. Hot water is let down upon the malt in the mash-tun when at the proper temperature; and in order to adjust this the foreman of the brewhouse ascertains, by the aid of a thermometer, the temperature of the water through a temporary opening in the upper part of the boiler. This is shown in Fig. 4, where is also represented a balance-weight and graduated scale, which, aided by a float on the surface of the liquid in the copper, indicates the depth of the water.

Fig. 4.



When the water has acted on the malt for a certain period, and been constantly stirred with it, the liquor receives the name of *wort*, and is allowed to flow through pipes out of the mash-tuns into a large cast-iron tank, or *underback*, measuring 24 feet or upwards in length, by 8 in width. This is merely a general receptacle for the wort, into which the latter is collected when the mashing is completed. *Cooling* is effected by various mechanical contrivances, which differ very greatly in different establishments. Large, open, shallow, airy rooms, called *coolers*, or *cooling-floors*, in which a thin layer of wort was cooled by the access of air on all sides, was the mode formerly adopted. This plan has, however, been superseded by contrivances, in which 100 square feet of surface is made to abstract as much heat as was formerly done by twenty times its extent. One of the *refrigerators* employed acts in the following way:—The hot wort is allowed to flow out of the underback into an oblong vessel, and out of this into another receptacle in the same part of the building. A continuous pipe, between 300 and 400 feet in length, passes backwards and forwards through the oblong vessel, and through this pipe cold water flows incessantly from an Artesian well 200 feet in depth. Constant currents of wort run in one direction through the apparatus, while a current of water in an opposite direction flows through the pipe which cools the wort.

Fig. 5.



The temperature of the wort may be cooled even to that of the water were it required, either by increasing its influx and retarding its efflux, or by permitting a larger quantity of water to flow through the pipe. The proper temperature is attained by regulating the relative streams of wort and water by suitably adjusted valves. Fig. 5 represents the refrigerator at the end where the wort enters, and where the water leaves the pipe, after having performed its office; collateral with the refrigerator is the under back. Not only does this method require much less room than that of the cooling-floor, but the refrigeration is greatly accelerated, and the manufacturer is rendered independent of the fluctuations of the weather; for since the water is brought from a source 200 feet below the level of the ground, it has, summer and winter, nearly the same temperature.

The wort produced differs from that made by the beer-brewer and distiller solely in possessing less saccharine strength. It suffers precisely the same process of fermentation.

From the refrigerator the cooled wort flows into the *jack-back*, a large circular receptacle sunk in the ground, whence it is pumped up into fermenting tuns. A valuable system of combination or centralization is observable in the arrangement of the conducting pipes. Large vessels are clustered, serving as a kind of common centre, from each of which openings lead to several other vessels, each orifice being regulated by a particular valve; for example, the liquid which, in various processes, is contained in the jack-back, has sometimes to be transferred to the fermenting tuns, at one time or other to a large back or cistern at the top of the building, and oftentimes to the copper; still there are not *three* openings from the back-jack for these several purposes, but *one*, which leads to a three-barreled pump, the barrels of which are respectively marked *tuns*, *back*, *copper;* so that, by turning one of three handles, the liquid can be conveyed to either one of these. Again, the back just alluded to is placed in connection with several other large tuns or backs, in different parts of the premises, to any of which its contents can be transferred by simply turning a handle. An hexagonal table is in one of the buildings, under the surface of which are six valves, all opened and shut by one key. On each tap the name of some particular vessel or building is inscribed, with which it is in adunation by an extensive series of subjacent pipes; and the overseer of this small piece of apparatus can control, in almost any direction, the flow of the liquid under manufacture.

The *gyle* is transferred from the fermenting tuns to other large casks, where it deposits, in course, a kind of acetous yeast—mother of vinegar; and being thence permitted to flow into the jack-back, it is drawn up one of the branches of the three-barreled pump into the large vat at the top; from this, as a centre, the gyle is allowed to flow into casks, where, after a longer or shorter period, it assumes the form of vinegar.

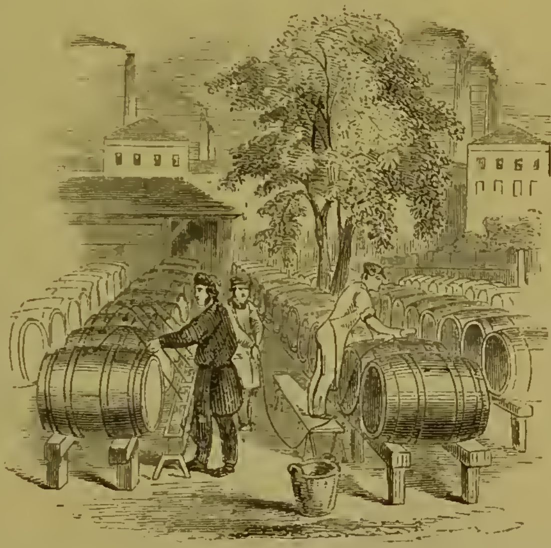
As before stated, transformation of the fermented wort into vinegar is effected at the factory in two ways, which are entirely opposite in their manner of operation. In the one case the casks containing the gyle are placed in close rooms, heated to a high temperature; in the other they are ranged in rows in an open field, where they remain many months. Different as these methods seem to be, yet the effect produced is precisely the same, namely, the conversion of the gyle into vinegar by the process of acetification, *i.e.*, the oxidation of the alcohol it contains into acetic acid. As regards the convenience and interests of the manufacturer, both methods seem to have their several advantages; for at many vinegar works both are followed, although one occupies a very much longer period of time than the other.

When *fielding* is resorted to, the gyle must be made during the spring months, and then left to finish during several months in the warmth of the season. The other process is technically called *stoving*, and in this case the casks containing the gyle are arranged conveniently in stove-rooms, and exposed to a certain temperature till the acetification is concluded. A cursory visit to one of these apartments readily convinces us of the progress of the manufacture by the pungent acetous odour of its atmosphere.

The fielding method requires a much larger extent of space and other utensils than the stoving, from the peculiarities always attendant upon it.

The casks, as already described, are placed in several lengthy parallel tiers, with their bung-side upwards, and left open. Beneath some of the paths which separate the rows of casks are pipes communicating with the "back" (vat) at the top of the brewhouse; and in the centre of each of these paths is a valve, opening into the concealed pipe. When the casks are about to be filled, a flexible hose is screwed on to this valvular opening, the other end of the hose being inserted into the bung-hole of the casks, and the liquor in the gyle-back at the brewhouse, by its hydrostatic pressure, flows through the underlying pipe and hose into the cask. The hose is long enough to admit of its reaching from the valve to all the casks in the same row, and is guided by a workman, as is seen in Fig. 6. After due time the vinegar is made, and is drawn off by the following ingenious operation. A long trough or sluice is laid by the side of one of the rows of casks, into which the vinegar is transferred by means of a siphon, whose shorter limb is inserted in the bung-hole of the cask. This trough inclines a little from one end to the other, and its lower end rests on a kind of travelling tank or cistern:

Fig. 6.

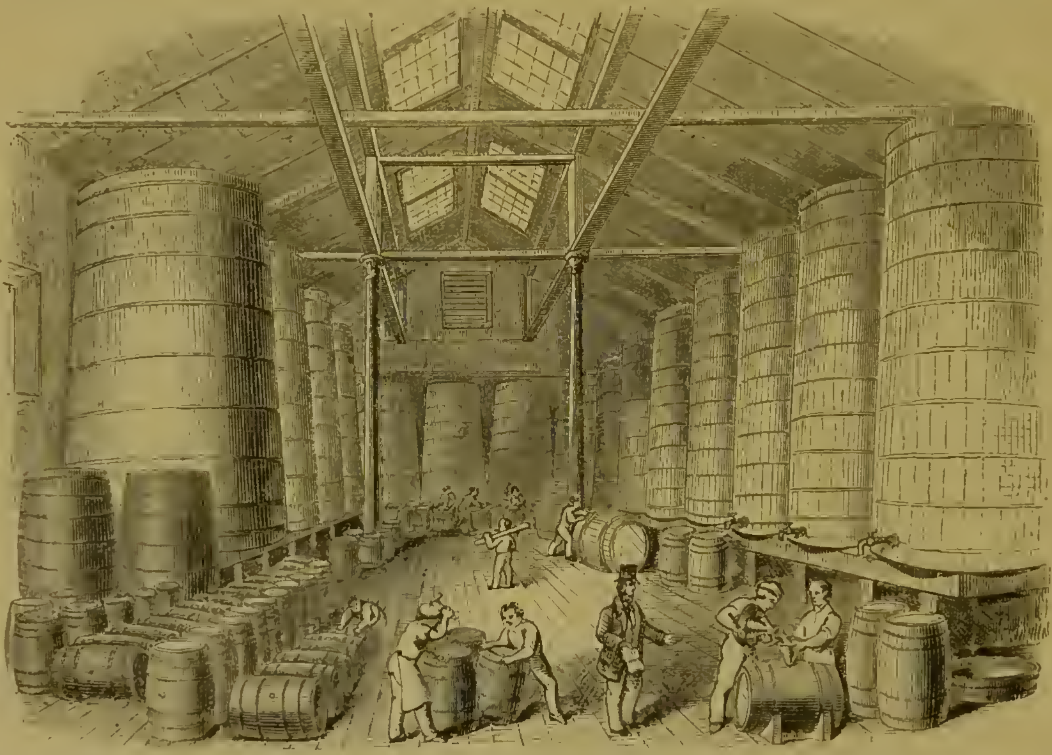


thus the vinegar from several casks is collected. A hose descends from the tank to the open valve of an underground pipe, which terminates in one of the buildings or stores. By means of steam power the pipe is exhausted of its air, causing the vinegar to flow through the hose into the valve of the pipe, and thence into the store-room. By this arrangement the whole of the vinegar is drawn off as it were invisibly. This arrangement is partly seen in the engraving. From the storehouse where the vinegar is received it is pumped into the *refining* or *rape* vessels, and filtered, to separate mucilaginous matter. These vessels are often filled with wood shavings, straw, or spent tanners' wood, but nothing acts so well in producing by filtration a clear bright vinegar as the stalks and skins of grapes, or raisins, technically *rapes*. The refuse of the British wine manufacture is very commonly employed.

It is a matter of so much difficulty to collect a sufficient stock of rapes to supply the filtering medium for large vinegar works, that, when once collected, no part of the materials relating to the factory are treasured with so much care.

Each "rape" or filtering vessel is fitted with a false bottom, on which the grape stalks are placed. Beneath the false bottom, and above the true one, a tap is inserted, which allows the vinegar to flow into a back or cistern. From this cistern a pump elevates the liquid to the top of the vessel. The filtering vessel is filled with vinegar, which filters through the raisin-refuse into the space beneath, from thence into the tank, whence it is pumped to the top of the vessel to recommence its circuit. Over and over again does this circuit proceed, the pump being kept constantly at work, and the vinegar incessantly in motion. The filtering substance gradually, but very slowly, wastes away, and is renewed from time to time.

Fig. 7.



By this process the last traces of alcohol become oxidized, all the coagulable nitrogenous matter and mucilage is removed, and the vinegar becomes transparent, or *bright*, as it is technically termed, and is then pumped into store-vats, where it is kept till required to be put into casks for sale. The "rapes" are immediately filled up with an equivalent portion of fresh vinegar, so as never to leave the raisin-refuse idle. The vinegar casks hold 116, 50, and 25 gallons, respectively. Each cask is examined and gauged before being brought into the *sending-out warehouse*, to see that it is sound and of proper dimensions. The warehouse is a large room, lined on all sides by store-vats, from which the casks are filled; after which they are finally coopered, branded, marked, &c., for the market.

The above description applies to pickling vinegar. Malt vinegar for household use is made in a somewhat different manner. The malt liquor is prepared in the usual way, at the temperature of 70° Fahr. (21° C.) and is then run into casks set on end, having each a perforated false bottom about a foot above the true one, upon which a layer of rapes has been previously placed. A small quantity of *argol*, or wine stone, the crystalline stony matter, chiefly tartaric acid, which is deposited during grape fermentation, is then added. After twenty-four hours the wash is racked off into another cask of the same description, and allowed to remain in this for a day or two, when it is drawn off into a third and fourth cask. After spending twenty-four hours in the last cask a portion of the liquid is racked off and supplied to the mothers. The remainder is then allowed to ferment quietly, as in the preceding instance, at a temperature of 70° Fahr. (21 C.). Argol communicates to it the appearance of wine vinegar. It is clarified by leaving it in the casks for some time with a little isinglass.

Pasteur in 1862 proposed a new method of making vinegar by the aid of the fungus *Mycoderma aceti.* A solution is first prepared suitable for the growth and propagation of the fungus, consisting of water with 2 per cent of alcohol, 1 per cent. of vinegar, and a small quantity of phosphate of potash, lime, and magnesia. The small plant soon covers the entire surface of the liquid, and at the same time the alcohol is acetified, upon which small quantities of wine, or alcohol and beer, are added until a sufficient bulk of liquid is obtained, upon which the charge is withdrawn carefully without disturbing the surface growth, and the apparatus used again as before. The vinegar thus prepared is said to resemble wine vinegar very closely in both aroma and flavour. The essential condition in Pasteur's process is careful attention to the growth of the plant. A vessel exposing 1 square yard of liquid surface, and capable of containing 10 to 12 gallons, yields daily 1 to $1\frac{1}{2}$ gallons of vinegar. The vessels preferred by Pasteur are shallow, and provided with lids; they may be square or round in shape; at opposite sides small holes are bored to admit air. The charge is introduced by tubes of gutta percha passing through the lids. These tubes are pierced laterally, beneath the surface of the liquor, with small holes. When the charge consists of wine or beer the fungi find sufficient nourishment; but when alcohol alone is used phosphates of lime, ammonia, potash, and magnesia must be added, so that the mother liquor shall contain about $\frac{1}{10000}$ of this mixture.

Sugar and Cider Vinegar.—In many factories, instead of a sweet wort of malt, a solution of sugar is often employed to produce vinegar. Several receipts are given for this department of the manufacture, the principal being the annexed:—Dissolve 10 lbs. of sugar and 6 lbs. of winestone (argol, *vide ante*) in 40 gallons of boiling water; put the solution into the fermenting tun, and when cooled down to 80° Fahr. (27° C.) add 4 quarts of beer yeast, and agitate the whole thoroughly. Grant the liquid repose for six or eight days at a temperature of 65° Fahr., till the vinous fermentation is ended; after which rack it off and submit it to one or other of the processes already mentioned, or by the *graduator* process, presently to be described. Another prescription is—

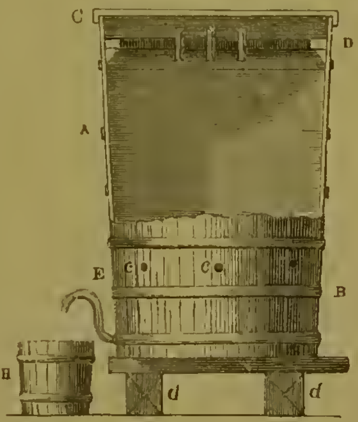
100 parts of water,	or	120 parts of water,
13 parts of brandy,		12 parts of brandy,
4 parts of honey,		3 parts of brown sugar, and
1 part of tartar,		1 part of tartar,

treated as before directed.

Quick Vinegar Process.—*Schnellessigbereitung,* German.—From the length of time necessarily occupied in making vinegar by the above methods, the name of *slow vinegar process* has been given to the manufacture since the discovery of a quicker mode of accomplishing the same end. Of the older methods, the only approximation to the process now to be considered was that of Boerhaave, already mentioned. Good vinegar is at present made from alcoholic liquors in the course of thirty-six or forty-eight hours. The slow and quick processes are conducted upon the same principle, namely, the oxidation of the alcohol; but the manner in which it is oxidized is different—the surface exposed in the quick method being many thousand times more extensive than in any former one.

When the oxidation of alcoholic solutions is slow aldehyde is the principal product. Aldehyde, however, absorbs oxygen with great avidity; hence, if the supply of oxygen be abundant, it is in the moment of fermentation converted into acetic acid.

Fig. 8.



In 1823 Schützenbach conceived the idea that by greatly enlarging the relative surfaces of contact of the alcoholic solution and air containing oxygen, the process of acetification would be greatly facilitated. His experiments proved successful, and soon after the German or quick vinegar process was generally adopted, especially in Germany and other countries where no duty is imposed upon alcohol.

The principle involved in the apparatus of course depends on an extreme division of the liquor being effected. This is very skilfully contrived. By making the solution percolate slowly through and diffuse over a mass of shavings, it forms a very thin liquid layer, the surface of which is exceedingly large, and is therefore better adapted for the chemical appropriation of the oxygen in the current of air which is transmitted over it. A gallon of liquor, if allowed to percolate slowly through the apparatus about to be described, offers a surface of about 100 square yards to the action of the air during its descent.

The vinegar generator, technically "graduator"

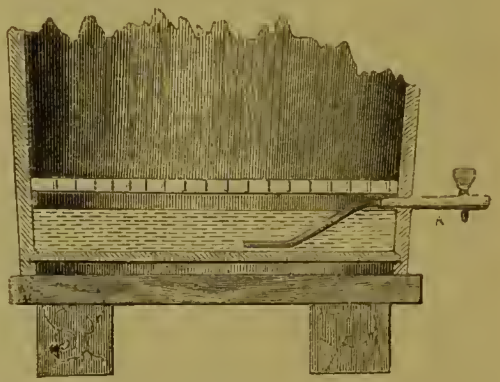
(Fig. 8), is a large tub or tun, A, of oak, of varying dimensions. In England the tun is often 13 feet high, 15 feet wide at the bottom, and 14 feet wide at the top; in Germany the size is usually much less, though the advantage to the manufacturer from the greater surface exposed in the large vessels is very great. The tun rests upon a stage, *d d*, of wood or brickwrok, 1½ foot high. A stout hoop of beechwood, upon which rests a perforated shelf, is fastened in the interior of the tub at B, 18 inches from the bottom, and 2 inch above this are eight or ten holes, *c c*, 1 to 1½ inch in diameter, bored at equal distances round the cask, and inclining downwards from the outside. Another strong beechwood hoop, D, is fixed 1 foot from the top of the tub, on which is placed a second perforated disc, pierced with holes 1 inch apart and ¼th of an inch in diameter. These apertures are loosely filled with cotton wick or packthread, a knot being made at the top end to keep them from falling through the cover. The threads pass down to the shavings, and serve the double purpose of conducting the liquor equally through the body of the tub, and also of stopping it from passing too rapidly through the tun. The space between the perforated shelves is filled to within a few inches of the top with shavings of deal or broken beechwood, which have been well washed with boiling water and afterwards stove-dried. Charcoal is sometimes used, and from its marvellous power of condensing oxygen in its pores, as well as the large surface it exposes, it is probably the best medium that can be employed. A thermometer is introduced a little below the top cover, the bulb of which reaches the middle of the apparatus, to indicate the rise or fall of temperature, as the subsequent oxidation of the alcohol is greater or less. Six larger holes are bored in the upper cover, 1½ inch in diameter, into which wooden or glass tubes, opening below it, and about 9 inches long, are adjusted; these serve as chimneys to carry off the deoxidized air from the vessel. A loose oaken cover, C, with a funnel opening in the centre, through which the liquids for charging the generator are supplied, protects the whole from dust. At 1½ or 2 inches from the bottom of the generator a pipe or glass tube, E, is inserted, which bends upwards nearly as high as the lower perforated shelf, and then curves downwards, so as to discharge the liquid when it rises so high as the shelf in the interior of the vessel, into an appropriate vessel placed to receive it. In the lower part of the vessel (Fig. 9) is fixed a wooden pipe, 12 or 15 inches long, and fitted with a plug or screw to act as a tap, by which to run off the dregs and other albuminous matters when they accumulate.

Everything being thus arranged, hot strong vinegar is poured through the funnel opening into the outer cover, and passed through the generator for one or two days, to sour the shavings and sides of the generator before passing the fresh spirituous liquors through for acetification.

To fill the tun a standard liquor is taken, consisting of 50 gallons of brandy or whisky, of 60 per cent. by volume, and 37 gallons of beer or malt wort, with about $\frac{1}{1000}$ of ferment. Acetification takes place slowly in the beginning; but as the shavings become gradually covered with the fungus of the *Mycoderma aceti*, or in the workman's phrase, impregnated with *mother of vinegar*, the oxidation is accelerated, and the larger the amount of the fungus the quicker the oxidation; so that the process goes on improving. The composition of the mixtures used varies very much; sometimes 5 gallons of the above liquor are mixed with 40 to 50 gallons of weak vinegar, and passed through the vessel at a temperature of 80° Fahr. (27° C.), and by this means the alcohol is more readily oxidized; or a liquor containing 1 part of alcohol, sp. gr. 0·850, 6 parts of water, and a trace of honey, yeast, or wort, may be used. According to Wagner, one of the mixtures very generally used is made up of 4½ gallons of proof brandy, 9 gallons of vinegar, and 26 to 27 gallons of water, to which has been previously added a liquid made by soaking a mixture of bran and rye meal in water, in order to promote the growth of the fungus *Mycoderma aceti*.

It is well known that essential oils, or a mere trace of wood vinegar, or any kind of tarry or empyreumatic matter, entirely arrest acetification; consequently, the vinegar used must be quite free from pyroligneous acid.

Fig. 9.



The tun being thus brought into a proper state of working, 15 to 20 gallons of the standard liquor previously mentioned are diluted with 60 gallons of soft water, and poured into the tun through the funnel in the outer cover, and permitted to pass through; it is again returned to it, unless there be several generators in the factory, in which case the liquor, after passing through the first, is allowed to percolate the second tub. Every succeeding hour 2½ gallons are drawn off from the second tub, that of the first being kept as vinegar, while the product of the second is always returned to the first vat or generator; thus, in twenty-four hours 30 gallons of vinegar are ready for sale: 150 gallons of superior vinegar can be manufactured daily in ten tuns, which one man can superintend. From the purity and clearness of the product, it resembles distilled vinegar; but to improve the colour and flavour, and render it more marketable, one pound of cream of tartar, and two pounds of brown sugar or molasses, is sometimes added to every 50 gallons, to suit the taste of the buyers. If honey or molasses has been previously added to the spirituous liquor, a fine coloured vinegar is at once obtained, hence this addition is often made for the sake of economy.

The temperature of the rooms should be 100° Fahr. (38° C.), and that of the standard liquor 125° to 130° Fahr. (52° to 55° C.) when poured in. After the working tuns have acquired a proper state for the acetification of the liquid, the charging liquid should always be run in at 78° to 80° Fahr. (26° to 27° C.), and the temperature of the room kept at 70° Fahr. (21° C.). During the time the solution is percolating the temperature of the generator rises to 100° to 108° Fahr. (38° to 41° C.), from the rapid oxidation of the alcohol, as will be indicated by the thermometer if the operation is going on favourably, so that if the quantity of liquor dealt with be large, it is not necessary to raise the temperature artificially. If a stronger acid be required than the product of the first and second vessels, the mode adopted is to mix the vinegar made in the first and second tuns with a stronger alcoholic liquor, and pass the mixture through a third tub; and if, when transmitted, it should be required still stronger, a fresh quantity of alcohol is added, and submitted to a fourth tub, to obtain acid of the strength required. The vinegar procured in this way, from the first and second tuns, should require 30 to 36 grains of pure potassium carbonate to neutralize the acid in every fluid ounce; that from the third tub, after being mixed with 20 gallons of the standard liquor, instead of 16 or 18, should neutralize 45 grains, and that from the fourth generator may be made to contain so much acetic acid that 1 ounce will saturate 60 grains of the pure alkaline carbonate.

When thick muddy liquors, or those containing much organic substance, such as beers or other mucilaginous liquors, are filtered through the tuns, their dregs deposit on the chips, the accumulation of which prevents the proper diffusion of the liquid through the generator, and consequently the complete oxidation of the alcohol is retarded. Should this happen, the chips are withdrawn from the generator, washed with hot water, and then after being steeped in hot strong vinegar, as in the forementioned instance, returned to the tub; or a stream of hot water, and afterwards a stream of hot strong vinegar, is made to pass through the tun without taking out the shavings; and afterwards hot strong vinegar, as before stated. It is better, however, always to charge the tuns with liquors free from sedimentary or slimy matters. Any such liquors ought to remain a sufficient time in the clarifying vessel to become bright before submitting them to acetification. Where these precautions are observed the tuns do not require frequent cleansing, and the products are purer and better. As before stated, many makers employ pieces of charcoal, about the size of a walnut, which have been deprived of their saline ingredients by dilute hydrochloric acid, and afterwards of the acid by water. The charcoal effects the oxidation of the spirit much more quickly than the shavings, and does not become so soon choked with slimy and other deposits: in a measure it also replaces the *Mycoderma aceti* by its condensing action upon the alcohol and aldehyde vapours and oxygen, and is itself competent to acetify the liquids, though not as energetically as spongy platinum and platinum black.

All the liquors spoken of in describing the *slow processes*, and indeed any alcoholic liquor free from empyreumatic products, may be converted into vinegar by this method.

The *wine malt* for charging the generators is made from wheat and barley malt, mixed in the proportion of 40 pounds of the former to 80 pounds of the latter. The whole is first well ground, then saturated with 40 gallons of water at 120° Fahr. (49° C.), and allowed to settle; the clear supernatant liquor is then drawn off. The residues are treated with water at 160° Fahr. (71° C.), agitated, and after settling, the clear solution drawn off as before: a final extraction of the soluble portions of the grain is made with water heated to 200° to 212° Fahr. (93° to 100° C.). The whole of the washings should not exceed 110 gallons. The solution is cooled to 75°, and 15 pounds of yeast added to it with much stirring, and the whole left at rest in an atmosphere of 80° (27° C.) for five or six days to undergo the vinous fermentation, after the termination of which it is ready for the generators.

Although this method is seemingly the perfection of rapid acetification in the vinegar manufacture, yet without proper care it is subject to many losses. In the slow methods, from the lengthy exposure to the atmosphere, a large quantity of material is evaporated and lost, even at the comparatively low temperature employed. The elevated temperature of the generators, of course, tends to increase the amount of this wasteful evaporation, and to completely carry off the alcohol and aldehyde; it has indeed sometimes happened that not a trace of acetic acid has been left at the termination of the process. When the quick vinegar-making process was first introduced great losses were experienced from the escape of the extremely volatile aldehyde (at that time unknown to chemists). Aldehyde boils at 68° Fahr. (20° C.). On this compound being discovered chemists at once saw that the cause of the deficiency was its imperfect oxidation, an evil immediately remedied by increasing the number of holes in the generator, so as to admit a larger supply of atmospheric air, in order that the whole of the volatile aldehyde might be converted into the more fixed acetic acid (which boils at 243° Fahr., 117° C.), before it has time to escape from the tun. This is one of the multitudinous instances of the advantages to be derived by the manufacturer having a knowledge of chemistry; and, moreover, is a happy illustration of the value of scientific investigation. All our records of the past show that every discovery of the chemist and physicist at some time or other plays its part in perfecting human civilization, however useless it may at first sight appear. The formation of aldehyde may be shown by closing some of the openings which serve to supply air to the tun; and when the alcoholic liquor has passed through, applying a solution of strong potassa to a portion of the clear liquid, upon which a brown resinous mass is obtained. This is one of the most characteristic reactions of this body. Another test for aldehyde is its power of reducing silver oxide to the metallic state. If some of the solution from the generator (when not doing its work thoroughly) be boiled in a test tube with a little oxide of silver, decomposition ensues; part of the oxide of silver is reduced to metal, and forms a brilliant and uniform coating on the glass; the solution remaining contains silver acetate.

The specific gravity of aldehyde is 0·790; it is a limpid, colourless liquid, very inflammable, and possesses a peculiar characteristic etherial odour.

From these facts it may be seen that the regulation of the supply of air to the vats is the most important of all the cares of the vinegar-maker.

If the supply be too great, the oxidation of the spirituous liquors will be too rapid, and from the consequent rise of temperature (probably to 120° Fahr., 49° C.) much alcohol will be lost. On the other hand, an insufficient supply of air stops the process halfway, and the manufacturer produces aldehyde instead of acetic acid. Theoretically, 1 per cent. of alcohol by weight should yield $1\frac{1}{10}$th per cent. of anhydrous acetic acid, or as much acetic acid per ounce as will neutralize 5 to 6 grains of pure potassium carbonate. In practice it is found that 200 gallons of spirit of 50 per cent. yield 1667 gallons of vinegar, neutralizing 32 grains of the alkaline carbonate per ounce, or 1775 gallons of 30 grains neutralizing power. According to theoretical calculations 1900 gallons of 30-grain vinegar should be obtained, which shows a loss in manufacturing of about 6 per cent. In many factories much of this loss is obviated by causing the vapours from the acetifying tuns to pass over a surface of cold water, in order to absorb any escaping alcohol or aldehyde. This water is afterwards used in extracting the soluble matters from fresh quantities of malt.

Some of the London vinegar works use a very large slightly conical tub or tun, 14 feet wide at bottom, 15 feet at top, and 13 feet high. Two and a half feet above the bottom of this tun a false one is laid; the space over this bottom is filled with coopers' wood shavings and chips, and the space beneath is destined to receive the liquor as it trickles down on the true bottom, in order to be pumped up in continual circulation. The reservoir of the wash is placed at a moderate elevation. The liquor discharges itself through a regulating stopcock or valve into a pipe which passes down through a suitable hole in the middle of the lid of the generator, and terminates a few inches under it, in a cross pipe shut at the ends, which is made to revolve slowly by mechanical power in a horizontal direction, round the end of the vertical pipe. This cross pipe is long enough to reach nearly to the sides of the tun, and being pierced with small holes in its under side, delivers the fermented liquor in minute streams equally all over the surface of the chips of wood. The wash falls thence into the lower part of the tun, through holes round the circumference of the false bottom; afterwards it is pumped up again, under certain modifications to be presently described. The air for oxygenating the alcohol into vinegar is supplied from two floating gasometers, which are made to rise and fall alternately by steam power. The ascending one draws its air from a pipe which passes into the centre of the tun, immediately under the false bottom, and as it redescends it discharges the air through a pipe into a cistern of water, which condenses and retains the alcohol vapour drawn off with the air. This water is used in making the next acetifying mixture. Fresh air is admitted into the top of the tun by the sides of the vertical liquor pipe, which is somewhat smaller than the hole through which it passes. Proper valves are placed upon the pipes connected with the gasometer pump, so that the air drawn off from the bottom compartment is prevented from returning. A small force pump is employed to raise the liquor continuously from the bottom of the tun to the cistern overhead. By this arrangement good vinegar is made in a few days without any perceptible loss of materials. The progress of the acetification in this apparatus is ascertained by testing the air for oxygen, as it is slowly drawn into the gasometers or expelled from them. For this purpose a bundle of twine, which has been impregnated with a solution of acetate of lead and dried, is set fire to and plunged into a bottle filled with the air, which should contain so little oxygen as to be unable to maintain the combustion.

By regulating the warmth of the apartment, the motion of the gasometer, and the admission of air, the due progress of the acetification may be secured. The vinegar has an average strength of 5½ per cent. of hydrated acetic acid, and is immediately ready for market.

Another process, not very unlike the preceding, patented by Mr. Ham, of Bristol, so far back as 1824, is still in operation at several works. The apparatus consists of a large vat, in the centre of which is placed a revolving pump, having two or more shoots pierced with holes, so as to cause a constant shower of wash—fermented wort—to descend from the top when they are set working. The lower part of the vat is charged with wash, the upper part with birch twigs, piled as high as possible, but without interfering with the revolution of the shoots. Between the surface of the wash and the joist which supports the birch twigs, a space of 3 or 4 inches is left unoccupied, and one or more holes perforated therein, in order to admit a current of air, either direct from the atmosphere, or from a blowing apparatus. If the wash be maintained at a temperature between 90° or 100° Fahr. (32° to 38° C.) and the pump kept in continual motion, a charge may be acetified in a period of two, fifteen, twenty, thirty, or forty days, according to the quantity of liquid and the mass of twigs through which it has to pass; but generally the birch twigs and liquid are so proportioned as to obtain the acid in fifteen or twenty days. The advantages offered by this arrangement are, that a wash made from raw grain with one-sixth of an admixture of malt will yield a vinegar equal to that from malt alone; besides this, any other liquor capable of fermentation and producing alcohol can be acetified as in the German process. The acetification can be arrested at any moment, and the current of air increased or diminished at will.

Messrs. NEALE and DUYCK, of London, patented a process for the manufacture of vinegar from beet-root in 1841. The method given by them is the following:—The tops and shoots of the beet are cut off, and the roots, after being thoroughly washed, are rasped into a fine pulp, with which a number of strong cloth bags are filled. These bags are placed in a powerful press, with a board or hurdle between every two, and subjected to pressure till the whole of the saccharine juice is extracted from the pulp. The strength of this juice varies from 7° to 9° of the hydrometer, and must be reduced by the addition of water to 5°. The liquid is then boiled for a short time and the *wort* removed to the coolers, in which it remains until the temperature falls to 60° Fahr. (45° C.). It is next conveyed to the fermenting vat, half a gallon of yeast being added to every 100 gallons of the wort. When fermentation has ended, the wash is pumped into the acidifying vessel, and is there converted into vinegar. The acidifying vessel consists of a strong vat, capable of containing 24,000 gallons, in the centre of which, a short distance above the bottom, a rose, or small inverted dome, is fixed, pierced with numerous small holes, and communicating by a pipe with a blowing apparatus. Upon the bottom of the vat lies a steam worm, one end of which is connected with a steam boiler, and furnished with a steam-cock, the other end being open to the atmosphere.

The interior of the vat is divided into several compartments by means of diaphragms or perforated false bottoms, and the cover of the vat is provided with a valve which opens outwards upon a very slight pressure from within. The vat is likewise furnished with a thermometer, the bulb of which is immersed in the liquid contained in it, by which the temperature of the liquid is known.

The mode pursued for converting the fermented wash into vinegar, by means of this apparatus, is as follows:—2000 gallons of vinegar are first let into the vat, to serve as *mother* to an equal quantity of fermented wash, which is introduced at the same time; and a little yeast being added, the whole enters quickly into the so-called acetous fermentation. After the action has commenced, air is forced into the apparatus by the blowing machine, which air, in its passage through the small holes in the false bottoms, is brought into intimate contact with the liquid, imparting to it a portion of its oxygen; the deoxidized air and carbonic acid evolved from the vinous fermentation being expelled through the valve in the cover, by the force of the current which is instituted through the vat. When the temperature, as indicated by the thermometer, falls below 70°, a current of steam is admitted into the worm by turning the cock, so as to maintain the heat of the vat at between 70° and 80° Fahr. (21° to 27° C.). By this means the liquid will in a few days be converted into vinegar; an additional 4000 gallons of the fermented wash are then introduced, and the preceding process continued; in a few days the whole 8000 gallons are converted into vinegar. Fresh charges are continually added and acetified, as just mentioned, till the vat contains 24,000 gallons of vinegar. When the acetous fermentation of the last charge has ceased, 8000 gallons of vinegar are drawn off, and fresh wort added, acetified, and drawn off alternately, about 16,000 gallons of made vinegar being always kept in the vat.

FRUIT VINEGAR.—Apples, grapes, and other saccharine fruits are expressed, the juice, with the addition of a little yeast, set aside in casks in a warm place—75° to 80° Fahr. (24° to 27° C.)—until the vinous fermentation has ceased, and then acetified by any of the preceding methods.

A very superior vinegar is made in Germany and other continental states from grape-sugar and the spirits produced from potatoes, beets, and molasses. The excise laws of England, however, prevent the adoption of these materials, except under heavy duties.

In some factories large quantities of sour ale and beer are converted into vinegar; but the product is much inferior to the vinegar made from wine or malt wort. The large amount of nitrogenous and other extractive matters which those liquids contain undergoes a second or putrid fermentation after the alcohol has been oxidized into acetic acid, and in doing so reacts upon the acid, leaving a liquid of a disagreeable odour, slightly resembling very stale beer. By addition of sulphuric acid this second fermentation is postponed for some time, but the vinegar has nevertheless a nauseous smell, which renders it objectionable.

Mr. J. C. KENT, of Upton-on-Severn, states that when he first undertook the supervision of the manufactory of Messrs. KENT & SONS, the firm was in the habit of buying beer, ale, and porter, that had gone hard and sour, for conversion into vinegar; and on one occasion the bankrupt stock of a large brewery at Cavendish Bridge, in Derbyshire, was bought. Murmurs had reached them on several occasions that the vinegar, on being kept, lost its acidity; but when the above large quantity of beer came to be sent out as vinegar the complaints became loud and frequent. It was found that the putrid fermentation of the beer was the cause of the mischief, and since that time no sour ale has been purchased by this firm. Some of the returned vinegar became exactly like vapid beer, less putrescent as to smell, but nevertheless in a phase of putrid fermentation. Mr. KENT also states that, in his opinion, it is impossible to make good vinegar from beer; and further, that although one or two manufacturers claim to be able to dispense with the addition of sulphuric acid to malt and grain vinegar, as mentioned further on, he has never been able to obtain a sample free from this acid.

Dr. STENHOUSE, in an investigation on sea-weed, has shown that when such bodies are caused to ferment in presence of lime, at the temperature of 96° Fahr. (35°·5 C.), acetic acid is generated in large quantities, and is, after subsidence of the action, found united with the alkaline earth in the form of acetate of lime. In three experiments with different varieties of sea-weed, he obtained as a result an average quantity of 1·8 per cent. of anhydrous acetic acid. He employed a temperature of 96° Fahr. (35°·5 C.), and added hydrate of lime gradually, keeping the mass slightly alkaline, till the fermentation ceased; after which the liquid was filtered off, evaporated to dryness, and the residue heated (to decompose the mucilaginous matters), when crude acetate of lime remained. This process has never yet been carried out on a manufacturing scale, although in some of the northern countries of Europe, as well as on some of the Scottish and Irish coasts, where sea-weed is plentiful, vinegar might be profitably produced.

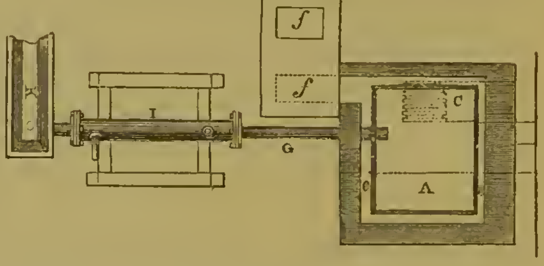
PYROLIGNEOUS ACID, or *Wood Vinegar.*—*Acĭdum Pȳrolignōsum*, Latin; *Holz-säure*, or *Holz-essig*, German; *Acide pyroligneux*, or *Vinaigre de Bois*, French.—The term pyroligneous acid is purely technical, and merely implies the crude acetic acid, contaminated with various empyreumatic oils, which is obtained from wood by destructive distillation. All organic bodies, except those which sublime without change, are decomposed when exposed to heat in closed vessels, their constituents interchanging with each other and forming new compounds, which are of sufficient stability to resist the particular temperature employed. Thus the elementary components of wood, after a certain amount of heat is applied, arrange themselves into combinations quite distinct from those in which they originally were. Some of these are gaseous; but at moderate temperatures by far the greater part are liquid, the quantity of the latter depending entirely upon the greater or less degree of heat applied in the distillation.

The main cause of decomposition of such an organic body as wood by heat is, that the strong affinity of its contained oxygen for carbon and hydrogen, and the comparatively great stability of the more simple compounds of these bodies, causes their formation immediately that there is a sufficient amount of commotion created amongst the atoms of the original body to allow them to commingle freely. Heat sets up the necessary amount of vibration, and those compounds are at once formed which can resist, without rupture of their constituents from each other, the multitude or amplitude of the vibrations corresponding to the temperature at which they are evolved.

As a general rule, those bodies containing much oxygen are decomposed at comparatively low temperatures. Acetic acid is an exception; as has been before observed, a dull red heat does not cause its constituents to fly sufficiently apart from each other to cause their total separation, and the compound therefore remains unchanged. To this circumstance is due the large amount of acetic acid which is produced during the destructive distillation of wood. The composition of wood may be taken as $C_6H_{10}O_5$ (or $C_{18}H_{30}O_{15}$) cellulose, also called lignin. The hydrogen and oxygen being in the proportions to form water, the withdrawal of carbon would form acetic acid thus:—$2C_6H_{10}O_5 - 2C = 4C_2H_4O_2$. As might be anticipated, acetic acid is amongst the earliest and most abundant products of the distillation of wood, and being volatile, escapes decomposition at the higher temperatures employed later. As the distillation progresses, marsh gas (CH_4), olefiant gas (C_2H_4), tetrylene (C_4H_8), and volatile oils, such as benzol (C_6H_6), toluol (C_7H_8), naphthalin ($C_{10}H_8$), paraffin ($C_{20}H_{42}$), phenol (C_6H_6O), &c., are given off. At the close of the operation nothing but charcoal remains. The actual facts which are observed in the distillation of wood are as follows:—First, the water passes off, which is extraneous to the wood; secondly, the wood itself is decomposed, and gives rise to water and the crude acetic acid, which is next eliminated; thirdly, condensable matters containing an excess of carbon, forming the tar and oily substance, pass over; and lastly, towards the close of the distillation, carbonic oxide and marsh gas are evolved, leaving in the retort a charcoal similar in form to the wood introduced.

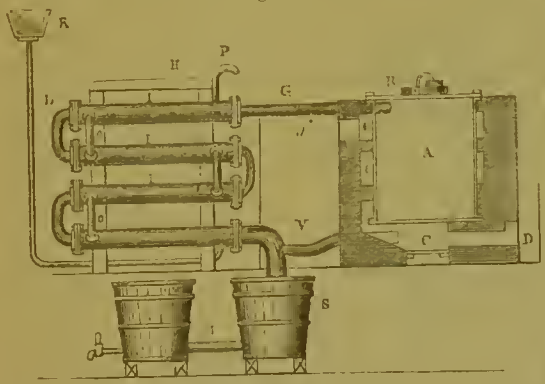
The distillation of wood is carried on in large cast-iron cylinders, or in square ovens made of stout sheet-iron, riveted firmly together, the heat being applied to them directly. A convenient apparatus for the distillation of wood is sketched below. Fig. 10 is the plan, and fig. 11 the elevation of the apparatus. In these figures, A is a box made of cast-iron plates, firmly bolted together, of the capacity of 100 cubic feet. This box is imbedded in brickwork. B is a cover on the upper end of the box, through which the charge of wood is introduced, another such opening being at the opposite side (not shown in the figures), and is made air-tight by a cover such as B. Through this second door the charcoal is withdrawn. C, the firebars; D, the firedoor, through which the fuel is introduced; *c c*, the spiral course of the flame round the box, and *f f*, the flue passing into the chimney.

Fig. 10.



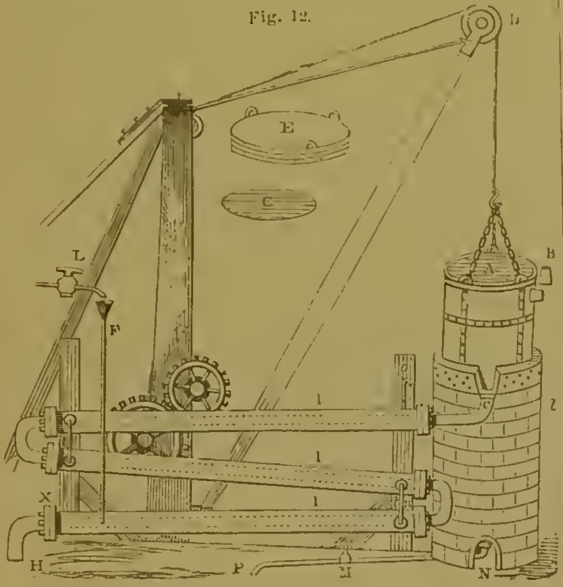
The iron pipe, G, conducts the gases and other volatile matters evolved to the condenser, which consists of a series of pipes, I, I, I, of large calibre, through which the pipe, G, passes, leaving a surrounding space through which cold water constantly flows. The pipes rest one above the other on a wooden framework, H. Through L a stream of water from the tank, K, enters the lower condensing pipe, and flows thence into the others by the connecting pipes, *o o*, till discharged by P, at a temperature reaching ebullition. Part of the distilled vapours and gases, in their progress downwards, condense into a liquid, which falls into the air-tight tank, S, and is thence conveyed by the connecting pipe, T, into another tank, collaterally placed, but at a lower level. V is a tube from the pipe G, projecting into the furnace, through which the uncondensed carbonaceous gases flow into the fire, where they are utilized as fuel. When the distillation has fairly commenced, these gases serve to maintain almost sufficient heat to effect the decomposition of the charge.

Fig. 11.



The usual time allowed for carbonizing each charge is twenty-four hours. After the wood is exhausted, the fire is withdrawn from the oven for six hours, so that the apparatus may cool, to allow the charcoal to be abstracted through the opening at the back, air-tight sheet-iron boxes being placed beneath the door to receive it—100 cubic feet of beechwood produce 60 per cent. of charcoal, as much brushwood being used to obtain the heat required to carbonize the wood.

Fig. 12.



The apparatus in use at Nuits and Rouen, in France, is the following:—In Fig. 12 A is a large sheet-iron cylinder with an opening in the upper part of one of the sides, into which an adapter, B, is introduced; C is the cover of the cylinder, fitted tightly to it with bolts and screws. The cylinder being charged with wood, the cover, C, is fixed tightly in its place, and the cylinder hoisted by means of a crane, D, fixed adjacent to the furnace, and deposited in an upright cylinder of brickwork which may be covered, at will, by means of a dome, E, made of brickwork. The cylinder is charged with wood and deposited in its receptacle, and the fire is then lighted. The tube, C, through which the distilled products pass off to the condenser, is next connected to the cylinder by means of the adapter, B, the joint being well luted. The condenser is similar to that attached to the oven at Fig. 11, and consists of a series of pipes, I, I, I, through which C passes. Water from the pipe, L, enters the pipes, I, I, I, by means of F and the connecting pipes, *t*, *t*, near the curvature, the heated water being discharged by the pipe, *o*. The condensed products pass off into the covered receiver. The uncondensed gases are conducted through the pipe, P, back to the furnace, where they are consumed. The pipe, P, terminates in a rose, N, similar to that of a watering can, a few inches above the floor of the ashpit. The gases are by this means regularly distributed through the fire. M is a stopcock, by which the quantity of gas entering the furnace is controlled.

In some factories different methods of condensing the products are adopted. In a few instances cold air instead of water is the medium by which the vapours are condensed. The evolved products of the distillation are in this case caused to traverse an extensive range of piping of large diameter, or are conducted through a series of casks which have been connected together by pipes. Water is, however, by far the most effectual mode of condensation, and is therefore generally employed. About twenty-four hours are usually allowed to work off each batch of wood.

SCHWARTZ' carbonizer is shown in Figs. 13, 14, and 15. Fig. 13 is a bird-eye view of the furnace, Fig. 14 a section of the elevation following the lines *d d*, and Fig. 15 another section, following the lines C C. In each of these figures similar letters are used to distinguish the same objects. A A is the space where the wood is carbonized; *b b b b* are the apertures through which the wood is introduced and the charcoal afterwards is withdrawn; C C are the fires which heat the furnace; *d d*, the openings through which the acetic acid and the smoke, carbonic acid, oleaginous, tarry matters, &c., pass off through the pipes, *g g*, and thence through the condenser into the chimney; *e e* are crooked pipes descending from *g g*, which convey the tar condensed in *g* into the vessels, *f f* (Fig. 14); the bend in the pipes, *e e*, is made in order to prevent access of air into the apparatus; H H H H are wooden canals, wherein the acetic acid and volatile oils condense; I is the chimney, and *k* a small opening in the chimney, in which a fire is occasionally lighted to establish a draught. The furnace walls are of fire-brick, or they may be made hollow, the inside layer being fire-brick, and the intervening space being filled with aluminous earth and sand.

The furnace is first charged with the heaviest blocks of wood; between these smaller wood is then introduced, for the purpose of making the interior more permeable to the action of the fire. All the orifices of the furnace are then closed, and the fires at C C lighted, the current of air being instituted in I. as above stated. The blaze of the fire traverses the furnace and carbonizes the charge of wood, and the smoke and other vapours from the furnace pass by the exit pipes, *d d*, into *g g*, whence they escape into the condensers, H H, and thence to the chimney, I. The charge is known to be completely carbonized when the smoke issuing at I, which is at first black and heavy, becomes bluish and light. The chimney passage is then closed, and the opening of the pipes, *d d*, stopped up with wooden plugs, and then well luted with some plastic clay; the fire-doors are closed and the furnace left to cool. At the end of the second day two holes in the top of the furnace, which had hitherto been closed air-tight, are opened, and water introduced to extinguish the red-hot charcoal; the openings are again closed for a longer period, and when the furnace gets a little colder more water is added. So long as any red sparks are observed, the opening and pipes must be kept carefully stopped up, since the formation of a current of air would occasion the combustion of the charcoal, and consequently lessen its produce. After complete cooling the charcoal is raked out through the apertures, *b b*, and another charge introduced. The principle of carbonization in these kinds of furnaces is different from that in those already mentioned, inasmuch as in those first described the blaze from the fire never comes in contact with the wood, while in SCHWARTZ' furnace the blaze penetrates the entire furnace. The wood is carbonized with great rapidity by this arrangement. Another advantage which this furnace possesses is, that small wood may be burnt in C C; the acid and other valuable products being likewise collected in the condensers, H H. That which particularly distinguishes SCHWARTZ' furnace from that of LACHABEAUSSIÈRE is that no air can enter it except through the fires, C C, and that there is no loss from the combustion of charcoal. The cost of this furnace is about £120, the capacity being nearly 6000 cubic feet. The successful working of SCHWARTZ' furnace depends entirely on the exclusion of oxygen from the charcoal, a sufficient supply only being admitted to carbonize the wood.

Fig. 13.

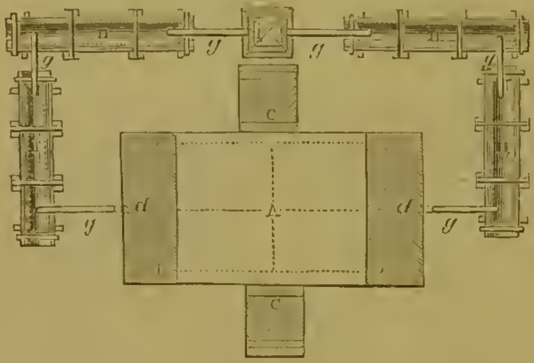


Fig. 14.

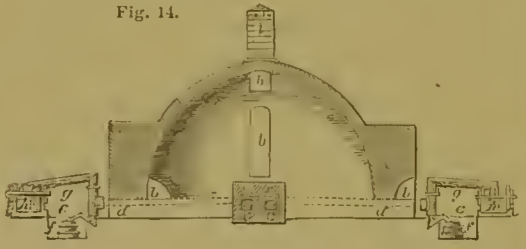
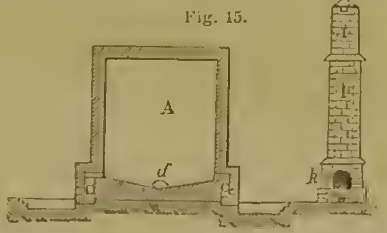


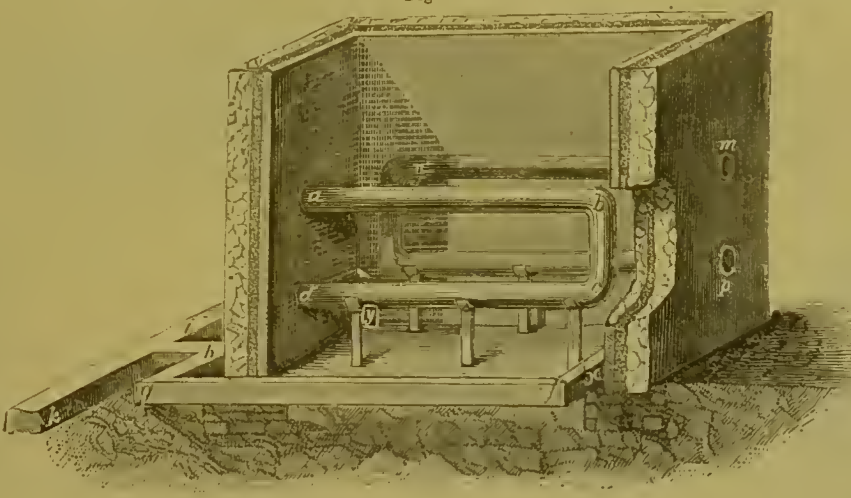
Fig. 15.



REICHENBACH'S carbonizing furnace consists of a square oven, the interior lining (represented in Fig. 16) of which is composed of firebrick and the outer case of any ordinary material. This oven is heated by means of tubes which traverse from one end of the case to the other, and are seen in *a b c d*, *m n o p*, in the figure; these tubes are from 1 to 2 feet in diameter. Heat is applied by lighting a fire in the tubes at *p* and *a*. When the temperature is raised so high as to cause the tubes to glow, the wood in the surrounding spaces of the oven abstracts the heat, and is thereby carbonized, the volatile products passing off at the bottom of the oven, through the opening at *x*, into the canal, *f g h*, and through that at *y* on the opposite side, into a similar canal. Both currents meet in the canal, *k i*, where the tar is partly deposited. From the tube, *k i*, the acetic vapours are carried off to the condenser, as in the ordinary process of the manufacture.

In England the retorts commonly employed are cylinders from 6 to 10 feet in length, and from 2½ to 4 feet in breadth. These are placed horizontally in brick-work domes. Their front and back ends are closed by doors which swing on stout hinges, or by plates screwed firmly to these openings. A pipe issuing near the extremity of the further end of the retort carries off the gases and condensable products to the refrigerator. A general view of the retorts and condenser is seen in Figs. 17 to 22. Fig. 17 shows the position of the cylinders when inclosed in brick-work, with the doors, K, closed. Fig. 18 represents a section of the cylinders. A, B, and C; the fire is at D, and the space, *c c*, shows the course which the flue takes round the cylinders till it reaches the chimney, E. The passage of this flue is more clearly seen in the plan, Fig. 20, where the dotted lines, *d d*, mark the circuits it makes. The spaces, A, B, and C (Fig. 20), indicates the cylinders and the pipes attached to them for conducting the distilled products to the tank, D, where most of the tar is deposited. From the tank, D, the product is conveyed through a small pipe into a condensing tube. This is more conspicuously seen in the elevation of the cylinder and tank, as shown in Fig. 21. Fig. 22 represents the section of a similar tank. The tank is protected by a cover, which fits into the enlarged part or groove at the top of the sides of the tank, acting as a water lute, as is seen at *h*. The pipe, *i*, carries off the acetic acid vapours to *k*, where they enter a serpentine pipe or worm in the large tub, C. A tub, *n*, is placed beneath the tank, A, to which it is connected by the tube, *m*, which penetrates through the bottom of the tank. The tarry matter, as it accumulates in the tank, falls through the pipe, *m*, into the tub, *n*. F is a tub placed below the condenser, C, where the acid is collected. The uncondensed gases pass off through the pipe, D, and are either consumed at *l*, so as not to corrupt the air in the factory, or are carried to the furnace.

Fig. 16.



The arc, B, Fig. 22, shows the position of the condensing vessel with regard to the cylinders, it being the same as F in Fig. 18. The space at H (Fig. 18) may be used as a drying chamber when preparing acetates. Sometimes two of these cylinders, placed in the same dome of brickwork, are heated by one fire, the flue playing round each, as in Fig. 23, where *a a* are the cylinders, *f f f* the fires, *c c c* ash-pits, and *o o o* the course of the flue to the chimney. As many as five such carbonizers are in some chemical works placed in one arch and heated by two fires. The cylinders vary in size in different works. In some works they are 9 feet long and 2 feet 10 inches in diameter.

Fig. 17.

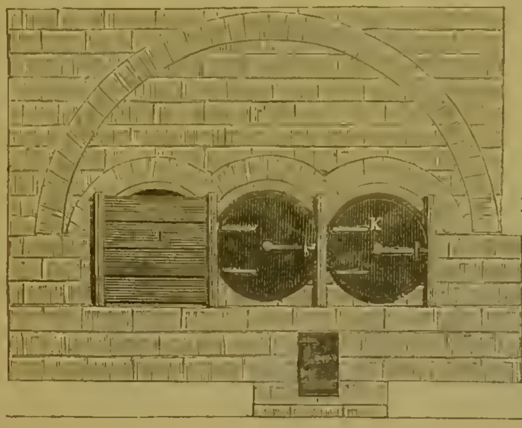
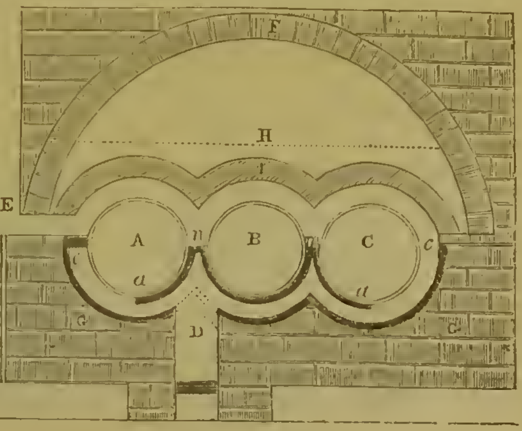


Fig. 18.



Thirteen of these are capable of holding 5½ *cords* of wood. Each cord is 16 feet 8 inches in length, and 2 feet 2 inches in breadth, and the same in depth, its weight being, according to the state of freshness or otherwise of the wood, between 23 and 25 cwts.: 1½ ton of coal serve to carbonize the whole. The usual time occupied by the carbonization of each charge is twenty-four hours. In other works the cast-iron cylinders are 6 feet long by 4 feet in diameter, each being capable of holding about 2 tons of wood, or ¾ths of a cord. The shape of the retort is not of much importance. Wrought-iron chests are found to be very effective; they are generally provided with an iron pipe 6 inches diameter, which passes up through the centre of the chest in order to convey the heat to the interior. Each chest is made to hold 1¼ cord of wood—160 cubic feet, a cord of wood being 128 cubic feet—which weighs about 3½ tons. Smaller boxes holding 1 cord of wood—their capacity being 108 cubic feet—are likewise used as carbonizers. Twenty-four hours is the time usually allowed to work off each charge; but if the demand for the distilled products be urgent the charge can be exhausted in sixteen to eighteen hours. In this case the products are less in amount than when the distillation is carried on slowly.

At works near Manchester the cast-iron cylinders are 6 feet long by 3 feet diameter, with square doors which hang on stout hinges. Generally 6 tons of wood are carbonized by 1½ ton of coal, one fire being made to heat two such cylinders, placed adjacent to each other in the same arch.

In the forest of Dean large square ovens are built, each of which contains two sheet-iron boxes, 4 feet 6 inches by 2 feet 9 inches in interior measurement. In each of these boxes about half a cord of wood is placed, and as the wood is piled up above the top of the sheet-iron box, the ovens are required to be of a size proportioned to this height. The doors of the ovens are on hinges, which are fastened and luted, when the charges are inclosed in the usual manner. These ovens are charged once a day; and as the wood is light twenty-four hours are more than sufficient to effect its carbonization. The boxes, when charged, are run into the ovens along a railroad, and are withdrawn in the same manner, whereby much labour is saved.

Fig. 19.

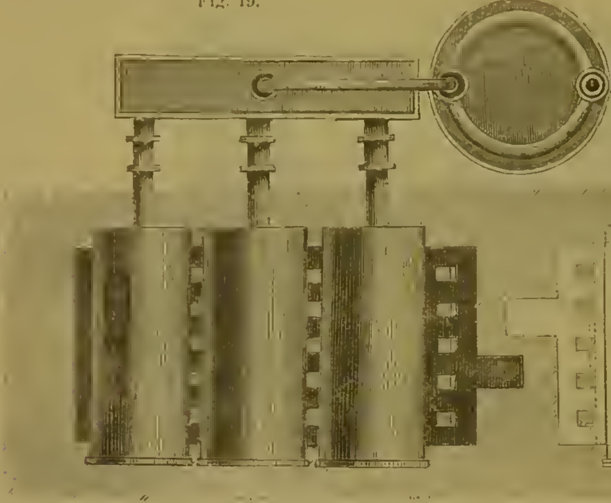


Fig. 20.

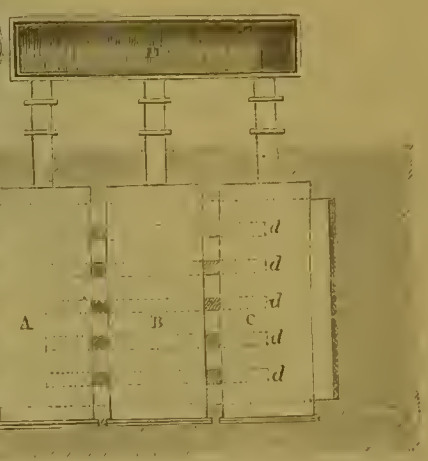


Fig. 21.

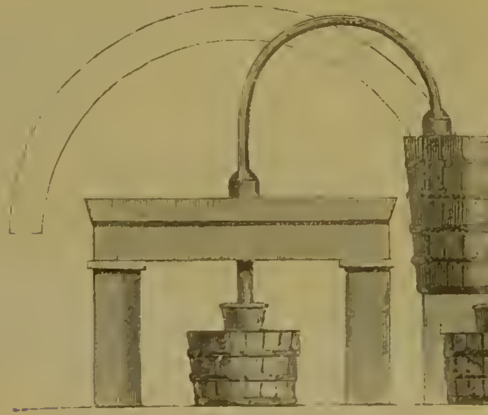
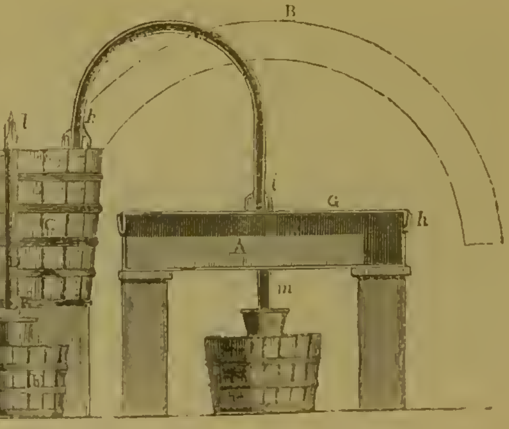


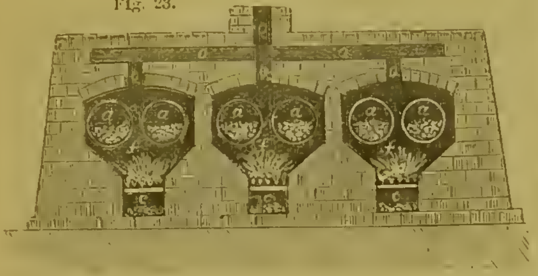
Fig. 22.



At Swansea they use a peculiar sort of apparatus—a large sheet-iron carbonizer, cylindrical in shape, and divided internally into six compartments, into which six sheet-iron vessels, 4 feet in height and 2 feet broad, fit. In the top of the carbonizer there is but one opening through which to introduce the vessels containing the wood; but in the bottom of the outer vessel there is a movable framework, by means of which each compartment is brought directly under the opening in the top of the carbonizer, and the charged box lowered into it by a crane. When the apparatus is fully charged the opening is closed by a sheet-iron door, which is bolted and then luted in the ordinary way, after which heat is applied. The sheet-iron case is imbedded in brickwork, and the fire placed directly under it, the flue rising spirally about the carbonizer. An apparatus similar to this is in use at Deptford.

It has been found that cylinders are the best ovens for the distillation of large billets, such as the heavy cord wood of Gloucestershire and the refuse ship-timber of Glasgow, Newcastle, and Liverpool—the most economical carbonizers being cylinders between 8 and 10 feet in length, and from 2½ to 3 feet in diameter.

Fig. 23.



Where light wood is used, such as that generally carbonized in the Welsh factories, the square ovens suit better; however, as the supply of wood is always subject to variation as to its bulk, it is frequently the practice to have both forms of apparatus.

The charcoal is at the end of the operation raked into sheet-iron boxes, or square pits sunk in the floor and lined with firebrick; both chests and pits are fitted with close-fitting covers, since, if air is not excluded, the charcoal, from its power of condensing gases in its pores, becomes so much heated as to take fire spontaneously. By shutting the charcoal in, the absorption is so far retarded as to keep the heat below the point necessary for ignition.

In many factories the charcoal is abstracted from the carbonizing cylinders by means of the following apparatus:—An iron diaphragm, almost the size of the interior of the cylinder, is placed near the mouth of the retort, having a chain attached to it which runs through the whole length of the carbonizer. The workman, by seizing this chain with a suitable instrument, draws out nearly the whole of the charcoal at once, and with less risk of breaking it than when rakes are employed.

The relative amount of charcoal, as well as of the gaseous and liquid products, depends on the species of wood subjected to distillation, and also upon the regulation of the heat by which it is effected. The temperature should at first be gentle, then gradually increase in proportion to the time, till towards the end it approaches an incipient red heat, in order to dissipate the whole of the volatile products of the wood. The strongest acetic acid is generally obtained from timber of slow growth on dry soils, the next quality from timber grown on moist ground, and the weakest, and that which contains most empyreumatic oils, &c., from pines and resinous trees. The produce of the last-mentioned variety is not, however, so inferior as is generally stated, when proper attention is paid to the regulation of the temperature and complete carbonization of the charges.

In establishing a chemical works for the distillation of wood, the first points to be considered are the supply of wood and the means of disposing of the charcoal. Charcoal is bulky, and deteriorates very rapidly in transit, and as a rule has a rather high tariff fixed upon it by railway companies. An abundant supply of water is also essential.

In this country the woods used for distilling are oak, beech, birch, thorn, crab, or apple, and exceptionally hazel, alder, ash, and maple. It is only under very special circumstances that coniferous woods are distilled. Holly and yew are highly esteemed when they can be obtained, but are not sufficiently common to be much used.

Of all these the oak is, according to both E. T. Chapman and Watson Smith, that which is most generally sought after, oak crop wood and old oak timber being the forms in which it is most readily obtainable; birch and beech follow next in order of superiority. The oak crop employed is almost invariably either the "lop"—that is, the lesser branches of large trees—or the saplings specially grown for their bark. In any case the greater part of the bark is removed before the wood is sold to the distiller. This "peeled oak," as it is called, would probably be used to the exclusion of every other wood, could it be obtained in sufficient quantity and at the same price as other woods; the only objection to it being that its charcoal is brittle, and therefore, in wood distiller's phrase, does not measure well. Charcoal *buyers* make no objection to it.

Beech is reported to be a particularly useful wood. It is the only wood employed in England in the form of large trees. Such timber is first sawn into convenient lengths for the oven, holes are then drilled in the logs, sometimes in the end, sometimes in the side, a charge of gunpowder introduced, and the log blasted to form suitable fragments. Beech wood furnishes much tar, gives a large yield of naphtha, and is said to be the best source of creosote; its charcoal is also good.

For the preparation of pure acetic acid, or a pure acetate, birch is beyond all doubt one of the very best woods, thorn and apple being nearly its equal. It is, however, by no means always the object of the distiller to prepare a particularly pure acetate; he generally takes into consideration the amount of yield of other profitable products.

Coppice wood, generally consisting of a mixture of hazel, alder, ash, and maple, together with smaller quantities of birch, beech, and oak, are very extensively used. These woods vary in value according to their size; they are most esteemed when of from fourteen to eighteen years' growth. In this country they are generally cut much too early, sometimes at the end of six years. According to German experience, the greatest yield of wood is not attained till much later.

Roots of trees are occasionally distilled, provided they are not decayed. They are broken up into moderately sized pieces, and generally furnish a very valuable distillate and leave good charcoal. The chief objection to them is the great labour required in breaking them up. They are sometimes put in the oven whole and heated for forty-eight hours.

As a general rule, hardly liable to exception, the larger the wood the better is it suited for distilling purposes, provided it can be completely charred in the twenty-two hours or so during which it is in the ovens.

Decayed woods of all kinds are to be avoided if possible, especially large logs of which a portion is decayed, as the charcoal from them generally proves to be spontaneously combustible. Holly wood also yields a charcoal which is very liable to spontaneous combustion.

The following is a tabular view, arranged by STOLZE, of the amount and strength of the products obtained from the distillation of several varieties of wood. The quantity of each kind of wood submitted to destructive distillation was *one pound*, a quantity suitable, in the generality of cases, to form a precedent for the manufacturer on the large scale:—

One Pound of Wood.		Weight of Acid.	One ounce of Acid neutralized of pure Carbonate of Potassa.	Weight of Empyreumatic Oil.	Weight of Charcoal.
	Botanical Names.	Ounces.	Grains.	Ounces.	Ounces.
White Birch	Betula Alba	7⅛	55	1⅜	3⅞
Red Beech	Fagus Sylvatica	7	54	1¼	3⅞
Large-leaved Linden	Tilia Plataphylla	6⅞	52	1¼	3⅜
Oak	Quercus Robur	6⅓	50	1⅓	4⅛
Common Ash	Fraxinus Excelsior	7¼	44	1⅜	3¾
Horse Chestnut	Esculus Hippocastanum	7⅜	41	1⅜	3½
Lombardy Poplar	Populus Dilatata	7⅜	40	1⅜	3¾
White Poplar	Populus Alba—abele	7⅜	39	1¼	3¼
Bird Cherry	Prunus Padus	7	37	1¼	3½
Basket Willow	Salix	7⅜	35	1¼	3½
Buckthorn	Rhamnus	7¼	34	1⅜	3½
Logwood	Hematoxylon Campechiaum	7¼	35	1⅓	2
Alder	Alnus	7⅜	30	1⅓	3½
Juniper	Juniperus Communis	7¼	29	1⅜	3⅝
White Fir	Pinus Abies	6⅜	29	2¼	3⅜
Common Pine	Pinus Sylvestris	6¾	28	1¼	3½
Common Savine	Juniperus Sabina	7	27	1¼	3⅝
Red Fir	Abies Pectinata	6⅜	25	2¼	3¼

Eighty-four Pounds of Wood.	Charcoal.	Charcoal per cwt. of Wood.	Acid liquor.	Sp. gr. of Acid liquor.	Sp. gr. of Acid liquor saturated with Lime.	Measures of Soda to neutralize Acid liquor.	Incondensable products.	Real Acid.
Birch,	23½	31·33	45	1·046	1·080	70	15	1·86
Elm,	21½	28·66	45½	1·036	1·075	83	17½	2·26
Willow,	18	24·00	49	1·029	1·045	29	17	0·77
Beech, low temp.	24	32·00	46	1·039	1·090	115	14	3·06
Do. high temp.	20	26·64	47	1·034	1·067	90	17	2·40
Laburnum,	20	26·64	46	1·030	1·055	75	18	2·00
Ash,	23	30·68	48	1·035	1·078	92	13	2·45
Alder,	20	26·61	48	1·030	1·065	70	16	1·86
Hawthorn,	20	26·64	27	1·040	1 100	140	37	3·73
Young Oak,	28	37·33	39	1·038	1·085	115	14	3 06

Three hundred and thirty-six Pounds of Wood.	Charcoal.	Charcoal per cwt. of Wood.	Acid liquor.	Sp. gr. of Acid liquor.	One ounce neutralized grs. of Bicarbonate of Potassa.	Dry Acetate of Lime produced.
Beech,	84	28·00	180	1·029	9½	25
Walnut	72	24·00	150	1·018	7	14½
Birch, cut three years	70	23·33	120	1·031	11	13
Oak,	91	30·33	190	1·022	8	24
Ash,	90	30·00	190	1·024	8	22
Apple,	70	23·33	200	1·017	6	18
Wych Elm,	70	23·33	180	1·018	8	16
Maple,	77	25·66	145	1 018	6	20

The average production of three works in the neighbourhood of Paris, from 4000 lbs. of wood, beech and oak, was:—

Charcoal,............1,014 lbs.
Acid Liquor,........2,335 " sp. gr. 1·027 = 416 lbs. of dry [acetate of lime.
Tar, ... 330 "

The following were the amounts obtained at the works of M. MOLLERAT, at Nuits, from 1000 stères of wood, weighing 5120 cwts.:—

Charcoal,................ 1,280 cwts.
Pyroligneous acid, ... 850 "
Acetic acid............... 54 "
Acetate of lead,.......... 152 "

In a well-conducted establishment in England the annexed were the quantities of crude acid liquor obtained by the distillation of 1634 cords of wood (each cord of wood weighing 512 cwts.), according to the longer or shorter time allowed to intervene between the cutting and the using of the wood:—

174 cords produced 23,923 gallons of about 10 lbs. each.
160 .. .. 27,720 .. .. ..
252 .. .. 30.424 .. .. ..
318 .. .. 40,584 .. .. ..
330 .. .. 55 900 .. .. ..
400 .. .. 50,700 .. .. ..

WATSON SMITH states that, according to his experience, the average yield which has been obtained, when old oak timber has been used in the retorts, after being split up and sawn into pieces of from 2 to 2½ inches long and 3 to 4 inches thick, was:—

Wood	Charcoal	Wood Acid	Tar.
From 1000 parts by weight, ...	327	509	55

The wood acid had a specific gravity of from 1·025 to 1·027 (= 5½° T.). In order to carbonize a ton of wood 10½ cwts. of coal were used on the average. He considers that the amount for fuel stated above, namely, 7 cwts. per ton of wood, can only be secured by the greatest economy and the most efficient flue and draught arrangements, so that the heat could be utilized to the uttermost.

The subjoined is one of the monthly statements of works in the vicinity of Paris, in which the wood is carbonized by utilizing the heat from ovens for the manufacture of coke.

Dr.	Frs.	C.
1000 Hectolitres of coal, at 50 frs. the 15 hecto.,	3,333	33
500 Stères, halfcords, of wood, at 9fr. 20c.,	4,600	00
Wages of coke-oven man,	120	00
Wages of six workmen,	450	00
Wages and lodging of two carmen,	200	00
Keep of two horses..	180	00
Salary of director of works,	200	00
Wear and tear, calculated at 10 per cent. on the value of the plant—60,000 frs.	500	00
5 per cent. interest on capital,	416	00
Rent of ground and cellars for charcoal,	100	00
Frs.	10,099	33

Cr.	Frs.	C.
1320 Hectolitres of coke, at 49 fr. the 15 hecto.,	4,312	00
450 Hectolitres small coke, at 32 fr. 50 c. the 15 hecto,	975	00
750 Loads of charcoal, at 6 fr.,	4,500	00
150 do., second quality, 5 fr.,	750	00
50 do., third quality, 4 fr.,	200	00
20 do., powder, 2 fr. 50 c.,	62	50
200 casks pyroligneous acid, at 15 fr.,	3,000	00
Tar,	165	00
Frs.	13,964	50

One franc equals 10*d.* English ; 100 centimes are equivalent to a franc. The French hectolitre is a measure of capacity, containing 100 litres, equal 22·009668 imperial gallons ; 4·5434579 litres equal an imperial gallon.

The temperature at which wood is distilled makes, according to CHAPMAN, a marked difference in the nature of the products obtained from it. In a series of experiments made on oak sawdust, he obtained from 13 to 27 per cent. of the sawdust in the form of charcoal, simply by varying the temperature at which the charring was effected. The higher the temperature the smaller the yield of charcoal. When, however, the heat was very gradually increased, so that the charring was effected at a low temperature, the yield was invariably only slightly less than it was when the wood had been charred at a permanently low temperature ; so that the large yield in the case of the low temperature was not due to imperfect charring.

When the wood is very rapidly charred the amount of liquid which distils from it is always less than when it is charred slowly, and the amount of uncondensable gas is very greatly larger. The amount of acetic acid is also larger at a low than at a high temperature. It is generally admitted that the largest amount of acetic acid is obtained with large ovens at a low temperature. On the other hand, when naphtha is desired, it is believed that small ovens very highly heated give the greatest yield ; though the large quantity of permanent gas generated must carry off with it much of the volatile spirit.

It is still a disputed point amongst distillers as to whether wrought or cast iron is the better material for the ovens. According to the experience of one manufacturer wrought-iron ovens will hardly last thirty charges ; according to another they will last five to six years. CHAPMAN considers that when properly protected by brickwork, and with careful firing, wrought iron is quite equal to cast iron in point of endurance. It is, of course, much less liable to crack, and may be made very much thinner, therefore wasting less fuel. Its disadvantages are, that vessels made of it easily lose their shape by overheating, and are somewhat apt to leak at the joints. Wrought-iron ovens are always furnished with cast-iron exit pipes and doors, as wrought iron is rapidly attacked by the acid vapours, unless it is sufficiently hot to prevent any condensation upon its surface. Wrought iron is only employed for the construction of cylindrical retorts.

Cast iron is most commonly employed. Its advantages are obvious ; the whole oven may be, and frequently is, cast in one piece, so that there is no chance of leakage from it. Its disadvantages are, that it is apt to crack with the fire, and when cracked it is difficult to repair it satisfactorily ; this is especially the case with cylindrical ovens cast in one piece. Square ovens are frequently built up of plates of cast iron bolted together.

The following are the dimensions of various ovens in actual use :—

	Description of Oven.		Length.	Diameter.	Depth.	Breadth.	Area.
			Feet.	Feet.	Feet.	Feet.	Cubic Feet.
1	Wrought iron,	cylindrical,	7·0	5·0	—	—	137·50
2	"	"	9·5	3·5	—	—	91·39
3	Cast iron,	"	10·0	3·5	—	—	96·00
4	"	"	10·0	3·1	—	—	115·40
5	"	"	4·5	3·0	—	—	31·77
6	"	"	7·0	2·5	—	—	34·30
7	"	rectangular,	7·0	—	4·50	3·66	115·29
8	"	"	9·5	—	5·00	3·66	174·00
9	"	"	10·0	—	4·33	4·33	187·76

Of these ovens, Nos. 1, 3, and 4 have been reported upon very favourably. No. 5 is an old pattern, not likely to be ever again constructed, though still in use. No. 6 is used to prepare special kinds of charcoal, and in some works to burn heavy timber from ship breakers' yards. No. 7 is not an approved pattern. No. 8 is considered to be a very good oven ; some ovens constructed on the same pattern have been in use for twenty years. No. 9 is the largest square oven which has been built.

Whatever the form of oven employed, it must be properly protected with brickwork from the direct action of the fire. No. 1 is protected for two-thirds of its circumference ; one fire heats two of these ovens. No. 2 is similarly protected, but each oven has an independent fire. Nos. 3 and 4 are only protected at the bottom ; they have a separate fire to each, though occasionally two ovens of the dimensions of No. 4 are heated by one fire. Nos. 5 and 6 have each a separate fire.

All the rectangular ovens have a separate furnace. As a rule, they are protected with brickwork as to their bottom plates only.

The composition of the permanent gas passing through the condensers, and which in this country is almost invariably allowed to escape, varies very greatly with the time that the wood has been in the oven and with the heat employed. If a sample of it be collected soon after the decomposition of the wood has commenced, it will be found to be colourless, have scarcely any perceptible smell, and to burn with a blue flame. It consists of a mixture of carbonic acid, hydrogen, and marsh gas.

As the temperature rises, the escaping gas becomes clouded with a tarry smoke; it now burns with a yellow smoky flame. If a sample of it be collected and allowed to stand for two or three days in a bottle, it deposits tar and becomes colourless; and if the temperature of the ovens has been kept very low it burns with a yellow, slightly luminous flame. It contains carbonic acid and gases absorbable by bromine. After these have been removed, the gas burns with a blue flame like that of alcohol, and consists of a mixture of carbonic oxide and hydrocarbon gases.

Towards the end of the operation the gases are richer in illuminating power, derived from gases not absorbable by bromine. Even right up to the end of the operation, however, the gas when purified by long standing, treatment with bromine, washing with alcohol, and then with aqueous potash, has very little illuminating power. It does not appear, therefore, that any hydrocarbons of the marsh gas series of at all complex formulæ are produced. Carbonic oxide increases in quantity as the distillation progresses; and at the end of the process, especially if the temperature be allowed to rise rather high, its quantity is very considerable.

The illuminating power of the gas, after standing and washing in alcohol, is greatest somewhat before the close of the distillation. If the temperature be kept low in the ovens, the illuminating power in the washed gas is never high. Of course the gas is charged with volatile liquids produced during the distillation, and therefore has considerable illuminating power as it leaves the condensers. The condensation should be so complete that no patch of condensed fluid is formed in the hand when the palm is held before the jet of escaping gas.

The termination of the distilling process is ascertained by handling the exit pipe from any of the retorts into the main; when this is found to have cooled down the distillation has ceased. The fire is then drawn, and the retort allowed to stand till the following morning to get cool. When the doors are opened the charcoal is raked out as rapidly as possible into capacious iron barrows, which when full are wheeled to the trap door, or man-hole of one of the underground strong tanks or vaults. The barrow with its load of still partially-ignited charcoal is upset over the open trap door, and the charcoal rapidly shovelled into the vault. When the charcoal from all the retorts requiring to be emptied has been collected in the vault, a few buckets of water are thrown in upon it, and the trap door is then securely luted down.

The mixture of tar and crude wood acid which came over into the condensers is then pumped into a large cistern at a convenient height above the ground and allowed to settle. The tar, of course, forms the lower, and the crude acetic acid the upper layer of fluid. When required, the tar is drawn off by a tap at the bottom, and the acetic acid liquor by a tap, or siphon arrangement.

Some years ago a patent was taken out by A. P. Halliday, of Salford, for the manufacture of pyroligneous acid, &c., from sawdust, spent bark from tanyards, and dyewoods exhausted of their colouring matters. His process proved very successful, and was adopted by the largest and most eminent manufacturers of decoctions in this kingdom. Ultimately the process was also carried on as an independent manufacture on a large scale.

The distillation of sawdust had for a long time previous been attempted, but without success. Ordinary ovens and retorts proved to be quite unfit for the decomposition of sawdust by heat. It was found that almost immediately heat was applied to retorts containing sawdust, it became coated with a close, hard layer of carbon, which, being a non-conductor, effectually barred the heat from penetrating to the interior of the mass, no matter how high the temperature to which it was exposed.

Halliday set himself the task of overcoming this difficulty, and Fig. 24 is a drawing of the ingenious apparatus which he devised for this purpose, and by which this obstacle was effectually overcome. The sawdust, spent dyewood, &c., are introduced into a hopper, H, placed above the front end of an ordinary cylinder, A A, in which a vertical screw or worm, C, revolves at such a speed as to convey the material in the proper quantities to the cylinder, which is placed in a horizontal position, and heated by means of a furnace, F. Another revolving screw or worm, B B, keeps the sawdust or other wood powder, introduced into the retort by C, in constant motion, and at the same time moves it forward to the opposite end of the retort. During their progress through the retort the materials are completely carbonized, and all the volatile products disengaged. Two pipes branch off from the extremity of the retort, one of which, D, passes downwards and dips into an air-tight vessel of cast iron, or a cistern of water, E, into which the carbonized substance falls; the other is an ascending pipe, K, and carries off the volatile products of the distillation into the condenser, which consists of copper or iron pipes immersed in or surrounded by water. The charcoal obtained, when ground finely, answers well for steel manufacturers and iron founders.

The quantity of acid obtained from spent dyewoods usually equals the amount obtained in the ordinary distillation of wood. It is, moreover, stated by Halliday, and also by Hadfield, that when burnt as sawdust in these retorts, the yield of acetic acid from pine and other resinous woods is very considerable. In ordinary ovens, as before remarked, these woods yield very little acid as compared with oak, beech, &c. It is also averred that in practical working eight retorts of the above description, 14 inches diameter, produce as much pyroligneous acid in twenty-four hours as sixteen retorts, 3 feet in diameter, worked on the old system, yield during the same time.

The average produce from eight retorts, taken for many weeks, carbonizing 22 tons of sawdust weekly, is the following:—

Pyroligneous acid 10° Twaddle = 1·05,..	2484 gallons.
Tar,..................................	240 "

To which, by way of comparison, is subjoined the average yield per ton of oak, when carbonized in the ordinary cylinders.

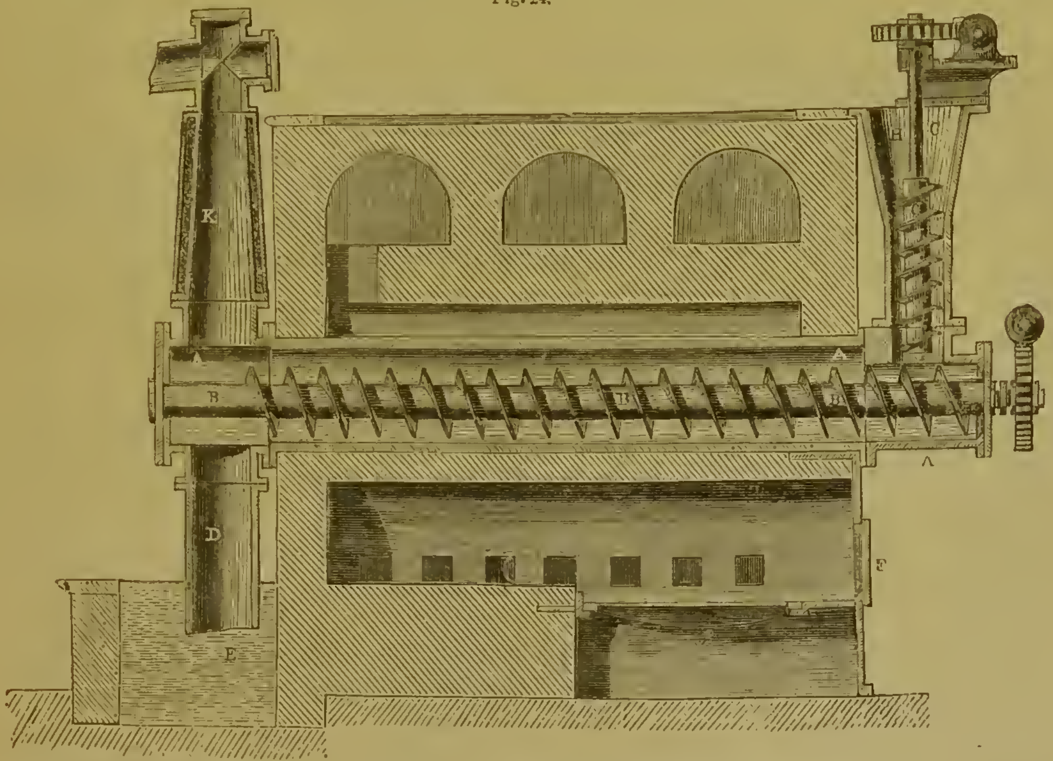
Weight of wood,..............		2240 lbs.
" pyroligneous acid,.....	1277 lbs.	
" charcoal, ...	600 "	
		1877 "
Loss—uncondensable gases,.........		363 "

According to the state of dryness of the wood when submitted to the retorts, the products are a little more or less; but generally the range of produce is between 124 and 127 gallons of 6° Twaddle, or 1·03 spec. grav., and 600 pounds of charcoal. It is therefore evident that the same quantity of acid is obtained from the sawdust, and is besides of a far higher specific gravity.

The average price of oak wood is eighteen to twenty shillings per ton, and for cutting this in convenient billets for the retorts and ovens the cost is usually about two shillings to two and sixpence per ton. From 7 to 10 cwts. of coals are found sufficient to carbonize 1 ton of wood. The tar obtained in distilling pine wood sawdust is of equal quality to that imported.

In the vicinity of Manchester, where exhausted dyewoods and sawdust are plentiful, a decided advantage in the yield and quality of the products is to be gained by operating upon those materials.

Fig. 24.



Many modifications of the HALLIDAY oven have been made, but all upon the same principle; namely, that of keeping the powdered wood constantly moving. One of the most ingenious of these is that devised by W. H. BOWERS of Manchester. His apparatus consists of a long rectangular retort, set at an angle of about 25° and heated by external flues, which run along its entire length. Within this oven are four revolving drums, moved by a small steam engine, which carry an endless chain to which projecting scrapers are attached at short intervals, in order to keep the floor clear of sawdust or charcoal. At the upper part of the retort a hopper is placed, through which the sawdust, wood-turnings, spent dye-wood, tan, or other materials, are introduced. The vapours are carried off by pipes in the usual way, and the charcoal after leaving the chain falls into a cistern, which forms the lower termination of the retort. The gases are prevented from escaping into the air by the thickness of the mass of sawdust at the top of the retort and by the water-joint at the bottom. The motion and the heat can be increased or diminished at will, the scrapers obviate the necessity of stopping working for cleansing purposes; the apparatus can therefore be worked continuously from month to month. Two of these ovens, with slow motion, produce 2500 gallons weekly.

SOLOMONS and AZULAY have patented a process, the main feature in which is the transmission of steam heated to a high temperature through the mass of material. The steam pipes are so arranged that every particle of sawdust is exposed to the superheated steam, and is thus completely carbonized. The products are those ordinarily obtained in ligneous distillation. The steam accompanying the distillates renders them dilute; this drawback is, however, compensated to a great extent by causing the mixed steam and volatile products to traverse a coil of piping placed in a pan of the distillates, from which the pyroligneous acid and other products pass to the main condenser. If this had not been suggested by a chemist, the patent could never have been profitably worked. The heated steam prevents the deposit of tarry and other resinous matters; consequently, no choking of the pipe need be apprehended.

The following brief sketch of the pyroligneous acid manufacture was contributed by JOHN RANDALL, manager of the Pitchcoombe Works at Stroud, to the last edition of this dictionary:—

"It remains still a disputed point whether small or large retorts are preferable. After a trial of different sizes, and some years' experience, the writer considers a retort of moderate dimensions the most convenient and serviceable. Those in use here are 9½ feet in length and 2½ feet in diameter, inclosed in brickwork, and placed horizontally, three or five in a set.

"They are secured in front by lids, fastened by means of a cross bar. At the back there is an exit pipe, 8 inches in diameter, connected with a main pipe. From this the liquor is conducted by a series of pipes, immersed in water, into a large tank. Retorts set in the method described are heated more economically, and the charcoal is good.

"Each retort holds about half a cord of wood, which, when of beech of the average dryness, weighs about 12 cwts. These retorts are charged once in twenty-four hours. As to the quantity of liquor produced from a given weight of wood, of course much depends on the condition of the wood, whether green or dry —that which has been cut down about six months is the best for practical purposes, the liquor being stronger in acid. A cord of wood in this state yields from 120 to 130 gallons of liquor, consisting of pyroligneous acid, water, naphtha, and tar, leaving charcoal in the retort equal in amount to about one-fifth of the weight of the wood originally employed. The next process is to separate these various products, and for this purpose the liquor is pumped up into copper stills, heated for safety by steam. Naphtha, in a weak and impure state, comes over first, then the pyroligneous acid, leaving a tarry residuum in the retort. Some manufacturers prefer adding lime to the liquor in the tank before it is transferred to the stills: this is, perhaps, best for the production of naphtha, but it involves the necessity of making black or brown acetate of lime, which from its inferior quality is often difficult of sale, except at a low price

"After distillation the acid is removed to large tubs or vats, and neutralized with lime. It is then allowed to stand for a few hours, and the clear solution siphoned off into evaporating pans. The vessels used here for this purpose are made of wrought iron, are oblong in shape, about 9 feet in length, 4 feet in width, and 2 feet in depth; they contain about 450 gallons. The solution is boiled down to the desired consistency, put into draining buckets, and then removed to a drying stove. This is the ordinary process; but when the acetate is required of superior quality, the solution is properly evaporated, then allowed to stand for eight or ten hours, carefully drained off from its sediment, and boiled to its crystallizing point. Simple distillation, though it separates a large portion of tarry matter, never renders the pyroligneous acid pure; this can only be effected by neutralizing the acid with carbonate of soda, evaporating the solution to dryness, and then subjecting the mass to fusion. The resulting *black cake*, as it is termed, is redissolved, boiled to a crystallizing point, and drawn out into large shallow vessels to deposit the salt."

Some time back PAUER discovered a method for the preparation of acetic acid and acetates from the distillation of wood, in which he dispensed with distillation, evaporation, and the greater part of the heating required in the processes generally pursued in this branch of the manufacture.

His method consisted in presenting to the acetic acid vapour, during the carbonization of the wood, a substance which can seize exclusively upon it, and thereby concentrate it.

The bodies which will the most readily satisfy this condition are those bases whose acetates are not decomposable at the temperature of the operation; such as potassa, soda, baryta, lime, magnesia, &c., or the carbonates of these bases, or any other salt whose acid can be displaced by the acetic acid. The author gives the preference, according to locality, to lime, a calcareous carbonate, magnesian carbonate, or carbonate of soda. The first three on account of their low price; the last because it yields the acetate of soda directly—a product prepared at a future stage for the entire purification of the acid.

PAUER'S process may be applied to any method of carbonization. The manner in which it is adapted to carbonization *en meules*—piles or masses of rough wood with earth—will now be described.

It is well known that carbonization *en meules* is effected by the heat produced by means of the combustion of a certain quantity of the wood of the pile when ignited. Orifices left at the foot of the pile give access to the air necessary for combustion; others pierced at different heights and in different positions by the workman charged with directing the progress of the carbonization, serve for the escape of the products of the combustion and distillation In these last orifices, at the point where the workman has judged it necessary to carry the draught, M. PAUER introduces tubes into the earth of about one inch interior diameter and half an inch in thickness, which, dividing over all the pile, terminate in bundles in numerous suitable vessels for collecting the products which are distributed around the wood stack. These pipes may be composed of several ends or short pieces, connected by joints, so that, when the progress of the carbonization requires the displacement of the orifices for the emission of the smoke, the extremity only of the tubes need be removed, thus avoiding the necessity of deranging the receivers.

The latter are generally simple casks, of 14 inches diameter and 3¼ feet in height, which are filled, in whole or in part, with pieces of lime, carbonate of lime, or carbonate of soda, divided into fragments varying in size according to the state of porosity of the particular substance used, interstices being left between the pieces to permit the passage of the vapours.

Acetic acid, and the other products which escape from the wood, are conducted by the pipes to the bottom of the cask, and traverse the different layers of carbonates or of lime which fix the acetic acid, whilst the other products escape at the top of the receiver.

It is proper to remark that this process presents one advantage over that by refrigeration, namely, that owing to the high temperature maintained in the interior of the casks, much less tar is condensed, and the subsequent purification of the acetic acid is thus rendered more easy.

The acetate of lime obtained in this manner may be submitted to the same operations as in the processes at present in use.

It is possible, under certain circumstances, to introduce into the midst of the pile, in the intervals left between the logs, the substance intended to unite with the acetic acid. Receivers are then unnecessary.

There are some factories which distil wood in close vessels expressly to obtain the acetic acid. For these the method described would have great advantages. In fact, there would be nothing to prevent the temperature of carbonization being so regulated that the acetate of soda produced in the apparatus itself should not be decomposed, at the same time keeping the temperature sufficiently high to destroy or expel the tar; the torrefaction would thus be combined with the carbonization, so as to obtain a concentrated and purified body at one operation.

Purification of Pyroligneous Acid or Wood Vinegar.—This is effected by saturating the crude liquor remaining after the spirit has been separated with a base, evaporating to dryness and calcining the dry salt, so as to decompose the oleaginous and tarry matters present, and afterwards distilling this body with an acid—sulphuric or hydrochloric acid—and rectifying the distillate repeatedly over chloride of calcium or carbonate of soda. Lime or soda is generally employed for this purpose. After the naphtha has been expelled the acid liquor is run off from the still into the tanks, to deposit a portion of its impurities, it is next pumped into another pan, and milk of lime, or burned lime, made into a thick paste with water, added slightly in excess, and the mixture boiled for a short time, and then run off into a vessel to rest for ten or twelve hours. During this time the excess of lime, and part of the impurities combined with it, precipitate, when the supernatant liquid is ready to be pumped into the evaporating pans. If the crude acid liquor is distilled before neutralizing with lime, the distillate is saturated with lime in an appropriate cistern or vat, and when the solid matters have subsided the clear fluid is pumped into the evaporating pans. In some works the evaporators are wooden vessels, lined with lead, in which is a coil of iron piping, through which steam passes, while in others they use shallow pans of sheet iron, having a fire beneath them. In any case the liquid is frequently stirred with a large wooden spatula, and the matters which rise to the top during evaporation are carefully skimmed off. As the evaporation of the liquor advances, the acetate of lime crystallizes and forms a layer on the surface, which is collected with large scoops or ladles, and deposited in baskets, supported on a frame, placed directly over the pans, in order to prevent the cooling of the drainings.

The whole of the acetate of lime is removed in the manner above described. When the crude acid is distilled previous to saturation with lime, the acetate of lime which it produces is called *grey acetate;* if the acid be neutralized without distillation the lime salt is called *brown acetate*, sometimes *black acetate*. The acetate is then heated in a drying furnace at a temperature of 450° Fahr. (232° C.), to carbonize the resinous and other impurities. To obtain the pure acid, the grey acetate of lime is dissolved in water till the solution has a specific gravity of 1·200; it is then filtered to separate the carbon, and sulphate of soda in powder added, after which the whole is briskly agitated, to insure the complete decomposition of the lime salt. The sulphate of soda is to be added until a small portion of the liquor gives no precipitate on the addition of a concentrated solution of this salt. The quantity of crystallized sulphate of soda required to effect a complete decomposition is about four times the weight of acetate of lime operated upon; that this large amount of the decomposing body is needed is owing to the formation of a double salt of calcium and sodium, thus:—

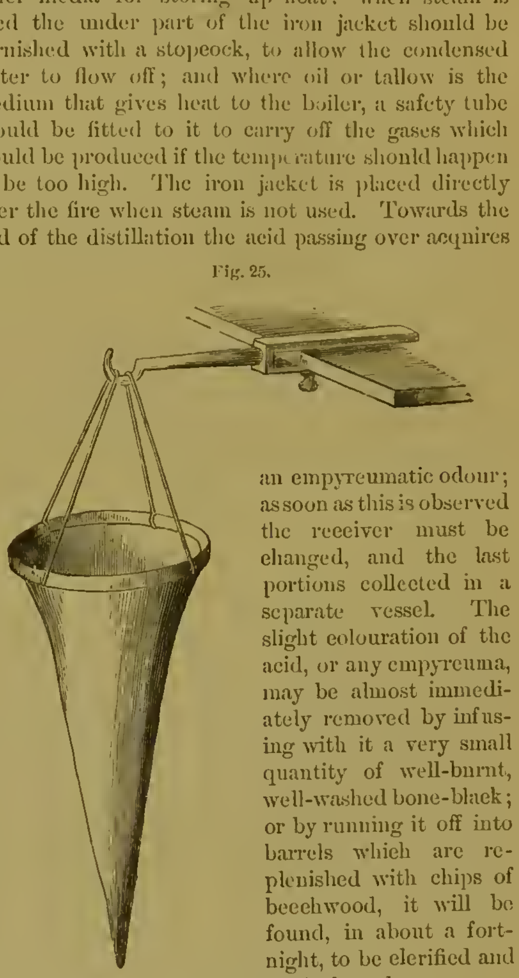
$$Ca(C_2H_3O_2)_2 + 2Na_2SO_4 = 2NaC_2H_3O_2 + CaNa_2(SO_4)_2.$$

The solution of acetate of soda, after the subsidence of the sulphate of soda and lime is drawn off and evaporated, and the precipitated sulphates are washed with water till the liquor is nearly tasteless; the washings being retained to dissolve fresh quantities of acetate of lime. The acetate of soda liquor is concentrated in pots 6 feet diameter and 3 feet deep, till it acquires a density of 1·300, care being taken to separate the impurities that rise to the top. If an excess of sulphate of soda has been used in the decomposition of the lime salt, it will now crystallize. The crystals are removed by the scoops or ladles, and thrown into a wicker basket above the evaporating pans, as before mentioned, so that the drainings may flow back to the remaining liquor without losing much heat. After separating the crystallized sulphate of soda the liquor is allowed to rest for eight or ten hours, to deposit impurities that are separated during the boiling, and is afterwards drawn off to the crystallizing pans, where it remains for three to five days. The crystals are then removed, and the mother liquor again concentrated to 1·300 (the tarry matters being removed as before), is again run off to recrystallize; and so on till the mother liquor no longer yields crystals. This liquor is evaporated to dryness and the residue calcined at a red heat, when carbonate of soda is formed, which may be extracted by solution in water; or the brownish residue may be calcined at 450° Fahr., and the acetate of soda dissolved out by water. The crystals of soda acetate are dissolved in fresh quantities of water, and re-evaporated till the solution is 1·500; left to repose for ten hours, the resinous bodies separated, and recrystallized at a density of 1·50: the mother liquor is treated as above detailed.

The crystals are next fused at a temperature of 400° Fahr. (204° C.), in an iron pot, and the mass kept constantly stirred till the whole of their water of crystallization is eliminated. During this part of the operation the greatest precautions are to be observed as to the maintaining of an even temperature—never exceeding 450° Fahr. (232° C.)—and guarding against any sparks coming into contact with the fusing mass. This latter accident would destroy the whole of the compound, since it burns like tinder in contact with fire. When white fumes rise from the liquefied salt it is an indication that decomposition is taking place, in consequence of the temperature being too high. The fire doors should in this case be immediately thrown open, and the fire itself drawn if the pot be still too hot. The termination of the drying is known by the subsidence of frothing, and by the fused mass presenting the appearance of an oily liquid, at which stage it is either drawn off by a pipe issuing from the bottom of the pot, or ladled out on iron plates to cool, and, when solidified, broken up into fragments. On dissolving this compound in twice its weight of water, filtering through bags similar to Fig. 25, and crystallizing, a very pure soda acetate is obtained. For the production of acetic acid from acetate of soda, a quantity of this salt is put into a stout copper still, and a deep cavity made in the centre of the mass, into which sulphuric acid, of spec. grav. 1·84, is poured, in the proportion of 35 per cent. of the weight of the salt; the walls of the cavity are thrown in upon the acid, and the whole briskly agitated with a wooden spatula. The head of the still is then luted and connected with the condensing worm, and the distillation carried on at a very gentle heat. The worm should be of silver or porcelain, as also the still head, and even silver solder should be used to make the joints in the body of the still.

In some factories it is usual to have the lower half of the still encased in an iron jacket, which receives high pressure steam, or holds oil, tallow, or other media for storing up heat: when steam is used the under part of the iron jacket should be furnished with a stopcock, to allow the condensed water to flow off; and where oil or tallow is the medium that gives heat to the boiler, a safety tube should be fitted to it to carry off the gases which would be produced if the temperature should happen to be too high. The iron jacket is placed directly over the fire when steam is not used. Towards the end of the distillation the acid passing over acquires an empyreumatic odour; as soon as this is observed the receiver must be changed, and the last portions collected in a separate vessel. The slight colouration of the acid, or any empyreuma, may be almost immediately removed by infusing with it a very small quantity of well-burnt, well-washed bone-black; or by running it off into barrels which are replenished with chips of beechwood, it will be found, in about a fortnight, to be clarified and ready for sale.

Fig. 25.



The acid which runs over is colourless, and contains about 45 per cent. of anhydrous acetic acid, its specific gravity being 1·05. Strong acid is procured by distilling the preceding acid, of specific gravity 1·05, with fused chloride of calcium, and receiving the distillate in a refrigerator; part of the acid which passes over crystallizes: the crystals are removed and dissolved in their own water, and subjected to a second rectification as before: this is continued till the whole of the acid crystallizes at 55° Fahr. (13° C.). These crystals deliquesce at the ordinary temperature, giving an acid of specific gravity 1·063, which is a monohydrate. If the acid, specific gravity 1·05, be required for culinary purposes, pickling, &c., it is diluted with about five times its weight of water to render it of the same strength as revenue proof vinegar; a little caramel—burned sugar—dissolved in water, and occasionally a little acetic ether, are added to confer a deep colour, and give it aroma.

When the acid is required for the fabrication of the other compounds used in the arts and manufactures where great purity is not essential, it is prepared directly from the crude lime salt by distillation with sulphuric acid. In some of the Welsh factories the following is the mode of working:—A cast-iron cylinder, 4 feet long and 2 feet in diameter, having one end closed tightly by a stout iron door, the front end being secured by a door firmly screwed on, which may be removed at pleasure, is provided; this door is divided into two parts, the upper part being two-thirds, and the lower one-third of the whole. Through the upper division a stout iron rod passes, and runs along the length of the cylinder to the opposite end, where it is fixed in a groove. From that portion of the rod which is within the cylinder numerous bars project at right angles, and extend through the whole concavity of the cylinder. A movable door occupies the lower division of the front of the apparatus, through which the contents of the interior are withdrawn, and the convex part of the cylinder is furnished with an opening, to which a door is screwed; the apparatus thus completed is termed an *agitator.* The cylinder is placed horizontally in brickwork, and the opening in the upper part affords the means by which the proper quantity of acetate of lime and sulphuric acid is introduced, in the proportion of 100 of the former to 60 of the latter. After charging the cylinder, motion is communicated to the axis passing through the upper part of the door either by machinery or manual labour. When the contents of the agitator are completely comminuted, it is then drawn off by means of the movable door in the lower part of the front end into a vessel placed below to receive it, out of which the half liquid or pulpy mass is ladled into strong cast-iron trays of 3 to 4 feet in length, 2 feet wide, and about 2 inches in depth: these trays are afterwards deposited in an oven 5 feet long, 3 wide, and the same in height, being separated from one another by iron rods laid between them, and on which they rest. A pipe passes off from the far end of this case, by means of which the gaseous acetic acid is carried to the condenser, which is generally a stout leaden pipe, placed in a stream of cold water. Fire is applied directly to the bottom of the oven, and continued till the whole of the acid is expelled. The crude acid is thus procured in large quantities, and is for the most part forwarded to London for purification. The principal impurities are sulphurous acid, sulphur, and traces of sulphydric acid, arising from the action of the carbonaceous matters in the salt upon the sulphuric acid; and resinous and tarry bodies, with a large quantity of colouring matters. From these matters it is freed by redistilling with a little bichromate of potassa, or bicarbonate of soda. This second distillation is effected in leaden vessels, encased by a cast-iron cylinder.

The ordinary yield of acetic acid from 1 ton of the crude acetate of lime, when treated with 12 cwts. of rectified sulphuric acid, specific gravity 1·84, or 15 cwts. of acid of 1·77 and 1 ton of water, is 1½ ton of rough acid, of specific gravity 1·05. Copper stills, or cast-iron stills, with flat earthenware or tin heads, often serve for the second distillation.

To obtain acetate of lime in a sufficient state of purity, Völckel saturates the crude acid with lime without previous distillation, whereupon a part of the resinous impurities separates in combination with lime, while the rest remains in the liquid, imparting to it a dark-brown colour; the liquid is clarified by filtration, or by simply leaving the impurities to settle down, and afterwards evaporated in an iron pot to about one half its bulk. Hydrochloric acid is then added, which causes the dissolved resin to separate, and also decomposes the lime-compounds of creasote and other volatile substances. The resinous matters and lime salts collect together in the boiling liquor, so that they can be easily skimmed off, and the volatile oils are expelled during the evaporation. The quantity of hydrochloric acid required for this purpose varies, of course, with the constitution of the wood vinegar, which again varies with the degree of moisture of the wood from which the acid is obtained; but the weight is usually from 4 to 6 lbs. for 150 litres (33 gallons) of the wood vinegar, or as much as will communicate a slightly acid reaction to the liquor. The solution of acetate of lime is then further evaporated, and ultimately dried at a high temperature to expel all volatile substances. Evaporation and drying are generally performed in the same iron vessel; but in operating on a very large scale, it is best to dry the salt on cast-iron plates; the last operation requires very great care. The volatile empyreumatic substances adhere very tenaciously to the acetate of lime and to the resin contained in it, and unless driven off by heat they pass over in the subsequent distillation together with the acetic acid, and impart to it a bad smell; the desiccation must therefore be continued till the acetate of lime becomes inodorous or nearly so; when thoroughly dried it has a dirty brown colour.

To separate the acetic acid, the purified lime-compound is distilled with hydrochloric acid. The distillation may be performed in a still with copper head and leaden condensing tube. If the operation be conducted with proper care, neither copper nor lead is found in the distillate. The quantity of hydrochloric acid required cannot be exactly given, because the acetate of lime contains variable proportions of foreign matters, such as resin and chloride of calcium. In general, however, from 90 to 95 parts of hydrochloric acid of specific gravity 1·16, will completely decompose 100 parts of acetate of lime, without causing the distillate to be much contaminated with hydrochloric acid. In any given case the quantity of hydrochloric acid required is easily determined by an experiment on a small scale. The apparatus may likewise be so arranged as to allow of the subsequent addition of hydrochloric acid, if the quantity first used be found insufficient. Whether the amount introduced is sufficient may be known by testing the distillate with nitrate of silver; so long as mere turbidity only is produced, the hydrochloric acid is not in excess.

Distillation of the acetic acid proceeds with ease and regularity. The acetate of lime dissolves in the hydrochloric acid, forming a dark-coloured liquid, while a quantity of dark resin separates. As the whole mass is liquid, the heat diffuses through it easily; and as the acetic acid passes over between 212° and 248° Fahr. (100° to 109° C.), and the acetate of lime has been already exposed in drying to a higher temperature, the distilled acid is but very slightly contaminated with the empyreumatic products which result from decomposition of the resin; moreover, the resinous matters, being lighter than the chloride of calcium solution, float on the top, and do not form hard incrustations in the still.

The distilled acetic acid has only a very slight empyreumatic odour. It is perfectly colourless, and if hydrochloric acid has not been added in too great excess, gives but a slight cloud with nitrate of silver. Any yellow tint that it may exhibit arises from particles of resin carried over mechanically; for the resin, separated from the acetate of lime by the hydrochloric acid, melts as the temperature rises, and forms a fluid layer on the surface of the chloride of calcium solution which is very apt to cause spirting; the resin should therefore be removed as far as possible before distillation, either by skimming it with a spoon or by filtration through a linen cloth.

The specific gravity of the acetic acid obtained by Völckel's process varies from 1·058 to 1·061, and contains more than 40 per cent. of anhydrous acetic acid. As, however, acetic acid of this degree of concentration is rarely used, and as a somewhat weaker acid is more easily separated by distillation from the solution of chloride of calcium, it is better to add a certain quantity of water either before or towards the end of the distillation. A good proportion is 100 parts acetate of lime, from 90 to 95 of hydrochloric acid, and 25 of water; this gives from 95 to 100 parts of acetic acid, of specific gravity 1·015. In this manner 33 gallons of wood vinegar will yield 60 lbs. of acetic acid of the above strength.

Acetic acid thus obtained may be still further purified by mixing it with a small quantity of carbonate of soda and redistilling.

The acid which passes over is free from hydrochloric acid and perfectly colourless, but still retains a slight empyreumatic odour; but this may be removed by distilling it with 2 or 3 per cent. of bichromate of potassa instead of carbonate of soda. Acetic acid purified with bichromate of potassa is, in fact, undistinguishable from that which is obtained from pure acetate of soda by distillation with sulphuric acid, or from pure acetate of lime with hydrochloric acid. It does not exhibit the slightest colour when heated with strong sulphuric acid, nor does it reduce even the merest trace of silver when boiled with nitrate of silver and ammonia. When saturated with oxide of lead it yields a white salt, the analysis of which agrees perfectly with that of pure acetate of lead. Peroxide of manganese may be used instead of bichromate of potassa; but the acid when thus purified gives, after a time, a slight turbidity with nitrate of silver. Any empyreumatic odour that it may retain can be removed by digestion with pure animal charcoal. As acetic acid is easily freed from hydrochloric acid, a slight excess of the latter during distillation is not injurious, but is, on the contrary, very useful in the after purification of the acid with peroxide of manganese or bichromate of potassa.

Rectification of acetic acid with bichromate of potassa, or with the peroxide of manganese, is generally conducted in a copper still with a leaden condensing tube. The only contamination of acid thus prepared is a small quantity of acetate of lead; and if access of air be prevented during the distillation, this impurity will be confined to the first and last portions of the distillate: by collecting these portions apart, to be used for the preparation of acetate of lead, the acid may be obtained perfectly free from lead. By observing these precautions the operator may dispense with the use of glass or silver heads and condensing tubes. The entrance of air into the condensing tube may be prevented by closing the end of the tube with a cork, through which is inserted a glass tube, bent in the form ∽.

The preparation of acetic acid by the method just described may be rendered simpler by subjecting the wood vinegar to a previous distillation, and thereby removing the greater part of the resin before forming the lime salt. But this distillation obviously entails increased expense for labour and fuel, because the same liquid must be twice evaporated; moreover, part of the acetic acid remains with the tar in the retort. On the small scale, the loss thus occasioned is unimportant, but in a large manufactory it would amount to something considerable in the course of a year.

When, however, the wood vinegar has been previously distilled a compound still is unnecessary. The vapour is then, instead of being condensed immediately, made to pass into a copper receiver, containing the quantity of lime required to saturate the acid, and is thereby completely absorbed. If the copper receiver be surrounded by some substance which is a slow conductor of heat, very little aqueous vapour condenses, so that the steam may be advantageously used to concentrate a solution of lime resulting from a previous operation. This process is, however, more complicated, and yields no more acetic acid than the simpler one first described.

The method here recommended is much cheaper, and yields a much purer product than the ordinary method of distilling impure acetate of lime with sulphuric acid; further, by the addition of hydrochloric acid during the evaporation of impure acetate, the volatile slightly acid bodies contained in the wood vinegar are removed, according to Schnedermann, more easily than by the use of solution of chloride of calcium, or by roasting the impure acetate of lime either *per se* or with hydrate of lime. In the latter process, even if it attains the desired end, a considerable loss is incurred from decomposition of the acetate of lime, inasmuch as that substance, from its infusibility, does not admit of any exact regulation of the heat.

The use of hydrochloric, instead of sulphuric acid, in the decomposition of the acetate of lime, has this great advantage, that the presence of resin, colouring matter, &c., in the acetate of lime, is harmless, provided that the salt has been sufficiently heated to drive off the free volatile substances. When, on the contrary, sulphuric acid is used, the acetic acid produced has always a bad odour, is saturated with sulphurous acid, and contaminated by a variety of products arising from the deportment of the resins at an elevated temperature. The sulphate of lime produced also forms a hard crust at the bottom of the retort, and in distilling on the large scale the under part of the alembic must be heated completely to redness to drive out all the acetic acid. The last portions of acid that pass over are moreover often turbid from sulphur, and the odour of sulphuretted hydrogen becomes perceptible, that gas arising from reduction of the sulphate of lime to sulphide of calcium at the bottom of the vessel; from these causes the cast-iron retorts soon become corroded.

Pure acetic acid may be obtained by the method above described at so very low price that it may be advantageously used in the preparation of acetates, especially of acetate of lead.

An important improvement in the purification of acetic acid was patented by H. B. Condy in 1868. It has long been known that a solution of acetate and chloride of calcium, in equivalent proportions, yields by slow evaporation large crystals containing $CaCl,CaC_2H_3O_2 + 5H_2O$, calcium aceto-chloride. H. B. Condy has found that, by judicious manipulation, these crystals are readily formed, and in a state of great purity, even when black and brown acetate of lime is operated on. From the pure double salt he obtains pure acetic acid by adding to it a strong acid and distilling.

For the manufacture of the calcium aceto-chloride for the subsequent manufacture of pure acetic acid, any inferior acid, such as that obtained by the distillation of commercial acetate of lime with sulphuric or muriatic acids, may be employed; but the most practical and economical plan is to take 8 cwts. of commercial acetate of lime, known as distilled acetate of lime, and dissolve it by boiling in 500 gallons of water. This is filtered or allowed to settle till the next day, when a quantity of dry chloride of calcium, equal in weight to seven-tenths of the acetate of lime employed, is dissolved in it by heat and agitation. If commercial chloride of calcium, or a strong solution of chloride of calcium, is used, it is necessary to take into calculation the water present, and to employ an additional quantity of chloride in compensation. This mixed solution of acetate and chloride of calcium, containing, in addition to the quantity necessary to form the aceto-chloride, an excess of about 20 per cent. of chloride, is brought by careful management when boiling hot, or nearly so, to the specific gravity of 30° Beaumé; if it should be of a lower specific gravity it is evaporated to this strength; if it be too strong it is diluted to the same point, and then drawn off to crystallize. When cold, and a crop of salt (aceto-chloride) has crystallized out, the mother liquor is drawn off and evaperated down till the hot liquor shows a strength of 31° Beaumé, when it is again drawn off to crystallize. These evaporations and crystallizations are repeated until three other crops of aceto-chloride are obtained. For the third crop the liquor drawn off boils at about 33° Beaumé, for the fourth at about 35° Beaumé, and for the fifth about 36° Beaumé. These five crops are of tolerably uniform quality, and leave by far the greater part of empyreumatic matter in the last mother liquor, which has given up nearly all its valuable and available acetate in the form of crystal, and is then either rejected, or it may be distilled with sulphuric or hydrochloric acid for producing an inferior acetic acid.

For manufacture of the pure salt these five crops, after they have been well washed and drained, are redissolved in twice their weight of water, the solution filtered through animal charcoal, and a further quantity of 10 per cent. or thereabout of chloride of calcium is added, after which the liquor is evaporated to 29° Beaumé, at which strength a good crop of white salt is obtained. The mother liquors from this salt are again evaporated down, and the hot liquor drawn off to crystallize at 30° Beaumé to obtain a second crop, at 31° for a third crop, and at 32° for a fourth crop. The mother liquor is then turned into the original solution of calcium aceto-chloride, from which the first of the five crops of rough salt referred to has been obtained.

Commercial brown acetate of lime, or even crude pyroligneous acid, may be employed for this process, but not with the same advantage. In the case of brown acetate of lime, it is necessary to observe that the weight of acetate of lime employed should be 20 per cent. more than that of "distilled" for a given weight of chloride of calcium, *i.e.*, about 12 parts brown acetate to 7 parts dry chloride; that the specific gravity of the liquor drawn off for obtaining each crop of salt (aceto-chloride) must be adjusted or regulated by experiment, since no two samples of brown acetate of lime are quite alike, and that it will depend in each case on the care with which the acetate may have been prepared. This can be ascertained during the process of evaporation by cooling and crystallizing a sample, after the boiling or hot liquor has attained 30° Beaumé. If after cooling a good crop of salt is not obtained, the liquor must be evaporated further until that object is accomplished. To obtain this salt by the employment of crude pyroligneous acid it is necessary to neutralize with lime, and to estimate the quantity of dry brown acetate it contains by evaporating a small portion to dryness and weighing the product. If the solution contains 1 part of brown acetate in 10 parts it will be simply necessary to bear in mind that 12 parts of the dry acetate in it require 7 parts of chloride of calcium, and that for the first crop it must be evaporated to 32° Beaumé; the tendency to crystallize must then be estimated by taking out a hot sample and allowing it to cool, and again evaporating until the required strength for crystallization is obtained. As a rule the mother liquors in all cases require to be evaporated to half their bulk to yield a further crop of salt, and then the tendency to crystallize can be ascertained.

When working upon commercial acetate of lime, the acetate is previously roasted so as to destroy some of its impurities. For this purpose a large iron retort is employed, 3 feet 6 inches in diameter, and 6 feet long; near the bottom a grating is so arranged as to touch the sides of the retort at a few points only, in order that conduction of heat from the sides may be but small. The acetate of lime is placed in sheet-iron trays about 2 inches deep, which are placed in the grating one over the other until the retort is filled; the trays are separated by iron rods laid in between them, and care is taken to keep them out of contact with the sides of the retort. The retort is then gradually fired until the outlet or head passing from the retort becomes heated by the escape of steam, after which the fire is steadily kept up but not raised higher until this escape pipe commences again to cool down, which indicates that the greater part of the water and the impurities have been driven off. The fire is now let out, and when cool, or nearly so, the charge is drawn. The operation generally takes about forty-eight hours for its completion.

When a solution of acetate of lime obtained by neutralizing impure acetic acid is operated upon, the aceto-chloride may be similarly roasted before redissolving and recrystallizing it. Roasting the aceto-chloride may also be done when working on commercial acetate of lime in place of roasting the acetate. Or in lieu of roasting, the original solution of acetate and chloride, or the acetate of lime alone, is sometimes filtered through animal charcoal, which has a remarkable affinity for, or power of, separating the empyreumatic impurities from the aceto-chloride, and also, though in a much less degree, from acetate of lime.

To make acetic acid, the calcium aceto-chloride, purified by crystallization, is decomposed with hydrochloric, sulphuric, or any other acid having sufficient affinity for lime to liberate the acetic acid contained in the salt, and the acetic acid separated from the chloride, and sulphate, or other calcium salt, by distillation.

The plan adopted is as follows:—To 112 parts of calcium aceto-chloride, 24 parts of sulphuric acid, spec. grav. 1·845, or thereabout, diluted with twice its volume of water, are added; these materials having been well mixed, are then distilled, and the acetic acid condensed in the usual manner. To render this acid quite pure, *i.e.*, free from hydrochloric acid, it is again distilled off alkali or acetate of soda, employed in the proportion of 1 lb. to 10 gallons of acid.

Another method is to take of aceto-chloride of calcium 100 parts, and of hydrochloric acid, spec. grav. 1·160, 50 parts each by weight, and after carefully dissolving the former in the latter, distilling off the acetic acid and condensing it as usual. This operation is performed in a suitable still, and in the manner usually followed in the manufacture of acetic acid by the decomposition of acetate of lime with hydrochloric acid. This acid is also redistilled with sufficient alkali to retain any slight trace of hydrochloric acid which may have passed over with the vapour in the first operation. The aceto-chloride is frequently sent into the market to be used by the purchaser as a convenient source of acetic acid. This process has the great advantage over many others that the preparation of the aceto-chloride can be conducted on the spot where wood is distilled, as for example, in foreign countries where wood is cheap, and may then be imported into this country for the process of producing the pure acetic acid to be completed.

A patent was taken out in 1873 by JOHN STEEDMAN, of Glasgow, for a process for the purification of acetic acid by passing the crude vapours through some hydrocarbon (preferably paraffin), kept at the temperature of the distillate, in order to separate the empyreumatic matters before condensation. The impurities are retained by the hydrocarbon, and the acid vapour passes on in a pure state to ordinary condensers, whilst the hydrocarbon is renewed periodically as it becomes charged with the impurities.

The process may be practically carried out in any convenient vessel or apparatus, in which the crude acetic acid vapours can be brought into intimate and continued contact with the purifying substance. Messrs. STEEDMAN & M'ALISTER employ a copper vessel of a rectangular form, about 5 feet long, 1 foot wide, and 2 feet 9 inches deep. This vessel is fitted internally with three partitions of copper or wood, which are horizontal in cross section, but slightly inclined longitudinally. The partitions are open at alternate ends, and the vessel being filled with paraffin, the acetic acid vapour, which is introduced from the usual distillatory apparatus by a pipe leading in beneath the closed end of the lowest partition, travels along through the paraffin from end to end beneath the partitions, and is finally led from the top of the vessel to ordinary condensing apparatus. The paraffin is kept sufficiently heated by a coiled steam pipe, or steam jacket, or in any other convenient way, and is withdrawn from the vessel whenever it is fully charged with impurities from the acetic acid. If wished, the acetic acid may be passed successively through two or more vessels containing the purifying substances. In practice it is found to be convenient to use the "heavy oil" for the separation of the grosser impurities, and to pass the acid afterwards through ordinary paraffin for further purification. A purifying vessel of the above dimensions is suitable for use in connection with a still of a size usually receiving about 100 gallons at a charge.

A large portion of the impurities taken up by the paraffin may be separated therefrom by agitation with hot water; and the paraffin may be further purified by the processes ordinarily employed for that purpose, and is then again fit for use as before. The heavy oil may be purified by ordinary processes.

Of late years woody fibre has been much used for paper-making and analogous purposes; various patents have therefore been taken out, having for their object the extraction of the acetic acid, methylic alcohol, resin, &c., without carbonization:

that of GEORGE FRY, in 1869, may be quoted as typical.

The first process is the cutting of the wood into strips or small pieces. The sizes of the slips into which it is preferable to cut the wood intended for paper-making are from half an inch to an inch and a half in length, and from one-eighth to one-quarter of an inch in thickness. If the slips are less than half an inch in length the fibre is liable to be too much cut and too short; and if they are more than an inch and a half in length, the boiling must be continued for a longer time, as the saturation of the wood takes place principally in the direction of the grain, so that the shorter the fibre the more perfect the saturation. On the other hand, it is found that a thickness of from one-eighth to a quarter of an inch is the most convenient size for the subsequent process of rolling and pressing to fit it for paper pulp. It is especially desirable that all the pieces of wood operated upon at one time should be of one uniform length and thickness, in order that the saturation of and pressure on all the fibre may be equal. The wood after being cut into suitable pieces is placed in cages (made of perforated zinc or copper wire) that exactly fill the inside of the inner cylinder of the boiler B (see Figs. 26 and 27). These cages are then placed in the boiler, the cover C is put on, and water is pumped into the boiler so as to fill it, with the exception of a small space left for expansion. By means of the steam jacket described below the boiler is then heated. The degree of heat that has been found most effectual for the purpose is that yielded by steam of 70 to 100 pounds pressure to the square inch, or equal to about from 300° to 330° Fahrenheit. This heat must be continued for from three to five hours, according to the nature of the wood and the size of the pieces. The heat employed may be greater, and if so the time of the boiling will be proportionately less, but that above indicated is preferred in practice.

Fig 26.

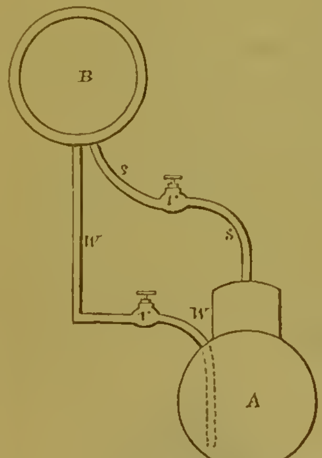
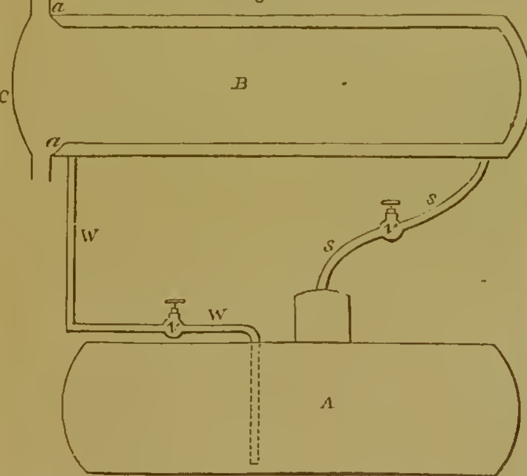


Fig. 27.



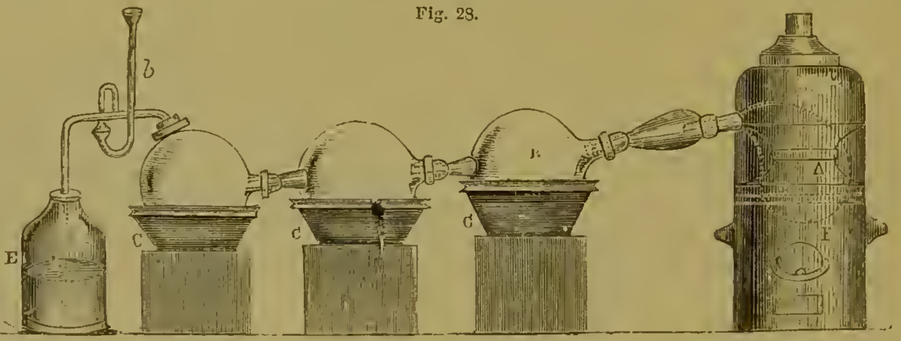
In the diagrams (Figs. 26 and 27) A represents any suitable boiler for generating high pressure steam; B is a boiler with a steam jacket, that is to say, it is composed of two cylinders of wrought iron rivetted together at *a*, *a*; C is a movable cover (to allow of the introduction and withdrawal of a cage containing wood) fastened to the flanged end of the boiler by means of stout bolts and nuts; S is a pipe for conveying steam from the steam chest of the generating boiler, A, to the space between the two cylinders of B; V is a valve which can be opened and shut at pleasure; W is a pipe dipping into the water of the boiler, A, down which the condensed steam returns from the steam jacket of B into A; V* is a valve so constructed that it is closed by pressure from A to B, but opened by pressure from B to A, by which means water can pass from B to A, but not from A to B. The cage or cages containing strips of wood being placed in the inner chamber of B, the cover C is bolted on and water is pumped into this chamber. The valve V is then opened. As soon as the pressure in the steam jacket of B approaches that in the generating boiler, A, the condensed steam (now water) passes freely from B back into A, and the contents of the inner chamber of B become rapidly heated by the steam supplied by the pipe, S, to the jacket.

The object of the peculiar arrangement of boiler referred to is to prevent the burning or charring of either the fibre or the liquid, to which it would be liable in ordinary boilers. When the wood has been sufficiently boiled the steam is shut off from the jacket of the boiler, and a valve opened in the pipe through which liquid is conveyed to and from the inner chamber of the boiler. The liquid in which the wood has been boiled then passes first through a coil of copper pipe placed in a cistern of cold water (renewed constantly during the passage of the liquid), and next through a box strainer of fine wire gauze into a still or retort. This still is heated in the usual manner by means of high pressure steam, and the liquid in which the wood has been boiled is distilled.

The first part of the distillate consists of dilute spirit (wood spirit, oil of turpentine, or other spirit, according to the kind of wood operated upon), and the latter part of dilute acetic acid. The spirituous liquid is rectified in the usual way, by which the greater part of the water is separated from the spirit. The acid liquid is run into an evaporating pan, where it is neutralized by an alkali, and then after evaporating the water by a gentle heat the acetate of the base remains. In order to obtain the almost anhydrous acid it is only necessary to mix gradually a strong acid (such as hydrochloric or sulphuric acid) with the acetate in sufficient quantity to take up the base, and to pour or distil off the acetic acid thus set free. The residuum (or solid matter left in the still) consists of rosin or resin, sugar, and a compound of iodine, the only part of which at present utilized is the resin. This is isolated by washing the residuum with water on a filter, the resin being insoluble in water, is precipitated in small crystals, from which the other matters being soluble are washed. As soon as pressure in the boiler has ceased by allowing the liquid to escape as above described, the cover C is removed, the cages are drawn out, and the wood fibre is passed between strong pressure rollers, or a hydraulic press may be used. The expressed liquid is passed into the still with the liquid obtained from the boiler. For the manufacture of paper the fibre is thrown into a washing or rag machine, and reduced to pulp. After washing it may be bleached as occasion may require by the usual process, viz., the action of chlorine, either alone or aided by an alkali or an acid. If the fibre is to be used for cordage the wood must be cut into strips of greater length, the boiling must be continued longer, and after rolling and pressing the fibre must be carefully washed.

Pure Acetic Acid from Brandy Vinegar.—By Völckel's process, brandy vinegar which contains from 12 to 15 parts of anhydrous acid is saturated with lime, and the solution strained through a linen cloth, and evaporated in an iron vessel. The dried salt is perfectly white. The decomposition of the acetate of lime is effected by hydrochloric acid, but the acetate of lime being less feculent than that obtained from wood vinegar, more hydrochloric acid is required for its decomposition. The amount used is about 130 parts of acid to 100 parts of the lime salt. Final purification is effected as usual.

Fig. 28.



Aromatic Vinegar.—Crystallized acetate of copper is the salt most usually employed: 20 lbs. of the powdered acetate of copper are introduced into an earthen retort of about 2 gallons capacity; the retort is luted and carefully dried before applying heat to distil the acid, and by this precaution it lasts much longer. The elongated neck of the retort is connected with a tubulated receiver; this is joined to several others, the last being furnished with a curved safety-tube, dipped into a vessel of water. The retort is inserted into a furnace, and a gentle heat applied, which is gradually increased; the rapid or slow development of vapour serving as a guide to direct the proper application of the heat. Fig. 28 shows the apparatus on a small scale; F is the furnace which heats the retort, A; B B B are tubulated receivers, immersed in cold water in the coolers, C C C; the last receiver, B, has a Welter's safety tube, *b*, the long arm of which dips into the water in E, where any uncondensed vapours from the receivers are absorbed. The fire in F is lighted, and the heat gradually raised so long as any vapours pass over; when no more vapours are given off, the distillation is terminated, the fire is withdrawn, and the apparatus allowed to cool: 20 lbs. of the copper salt produce, by distillation, about 10 lbs. of rough acid, of a greenish colour, and 1·061 spec. grav.; the residue in the retort consists of 6½ lbs. of metallic copper, mixed with a small quantity of charcoal. The crude acid is then rectified in a glass retort of the capacity of about 1½ gallon, to which is adapted a tubulated receiver, and heat applied by means of a sand-bath. A weak acid comes over first, which is collected separately until it acquires a density of 1·072; the receiver is then changed, and the distillation continued till the condensed products begin to become empyreumatic, when a third receiver is supplied to collect the last portions. An acid of spec. grav. 1·080 to 1·088 is found in the second vessel; the first and last portions are redistilled, and mixed with the stronger acid in the second receiver. The acid from 20 lbs. of acetate of copper, upon second rectification, yields 6 lbs. of acid, spec. grav. 1·085; 3 lbs. spec. grav. 1·042; and half a lb. of acid, spec. grav. 1·023.

Other metallic acetates may be used, but yield variable amounts of acid. The acetates of copper, silver, mercury, lead, &c., afford a larger proportion of acetic acid. Acetone, carbonic oxide, carbonic acid, and marsh gas, are evolved in various proportions; the metal of the salt is invariably left in the retort. The acetate of silver gives off no acetone when submitted to destructive distillation. Acetates, the metals of which retain carbonic acid at a red heat, produce chiefly the carbonate of the base and acetone, but very little acetic acid: the acetates of potassa, soda, and baryta behave in this manner; but when the metal cannot retain carbonic acid at a red heat, as in the case of the acetates of magnesia, zinc, and manganese, the acetone is accompanied by carbonic acid, and the oxides of such metals remain in the retort.

The small amount of acetone which passes over with the acetic acid, in the distillation of acetate of copper, imparts an agreeable aroma; and by the addition of a little camphor or essential oils, the *aromatic vinegar* of commerce is produced.

Acetic Acid and Vinegar Testing. — The specific gravity of vinegar is sometimes ascertained by a species of hydrometer, termed an *acetometer*. By dipping it into the vinegar under examination, and observing the depth to which it sinks, the number of the scale marking the level of the liquid indicates the density of the solution, from which, by means of a table, the percentage of real acid is ascertained. The results vary for different temperatures, as may be seen by the following table, compiled by Oudemanns, according to whom Mohr's determinations were made, not with pure glacial acetic acid, but with an acid containing 5 per cent. of water:—

Acetic Acid, $C_2H_4O_2$ per cent.	Density.		Acetic Acid, $C_2H_4O_2$ per cent.	Density.	
	15° C.	40° C.		15° C.	40° C
1	1·0007	0·9936	51	1·0623	1·0416
2	1·0022	0·9948	52	1·0631	1·0423
3	1·0037	0·9960	53	1·0638	1·0429
4	1·0052	0·9972	54	1·0646	1·0434
5	1·0067	0·9984	55	1·0653	1·0440
6	1·0083	0·9996	56	1·0660	1·0445
7	1·0098	1·0008	57	1·0666	1·0450
8	1·0113	1·0020	58	1·0673	1·0455
9	1·0127	1·0032	59	1·0679	1·0460
10	1·0142	1·0044	60	1·0685	1·0464
11	1·0157	1·0056	61	1·0691	1·0468
12	1·0171	1·0067	62	1·0697	1·0472
13	1·0185	1·0079	63	1·0702	1·0475
14	1·0200	1·0090	64	1·0707	1·0479
15	1·0214	1·0101	65	1·0712	1·0482
16	1·0228	1·0112	66	1·0717	1·0485
17	1·0242	1·0123	67	1 0721	1·0488
18	1·0256	1·0134	68	1·0725	1·0491
19	1·0270	1·0144	69	1·0729	1·0493
20	1 0284	1·0155	70	1·0733	1·0495
21	1·0298	1·0166	71	1·0737	1·0497
22	1·0311	1·0176	72	1·0740	1·0498
23	1·0324	1·0187	73	1·0742	1·0499
24	1·0337	1·0197	74	1·0744	1·0500
25	1·0350	1·0207	75	1·0746	1·0501
26	1·0363	1·0217	76	1·0747	1·0501
27	1·0375	1·0227	77	1·0748	1·0501
28	1·0388	1·0236	78	1·0748	1·0500
29	1·0400	1·0246	79	1·0748	1·0499
30	1·0412	1·0255	80	1·0748	1·0497
31	1·0424	1·0264	81	1·0747	1·0495
32	1·0436	1·0274	82	1·0746	1·0492
33	1·0447	1·0283	83	1·0744	1·0489
34	1·0459	1·0291	84	1·0742	1·0485
35	1·0470	1·0300	85	1·0739	1·0481
36	1·0481	1·0308	86	1·0736	1·0475
37	1·0492	1·0316	87	1·0731	1·0469
38	1·0502	1·0324	88	1·0726	1·0462
39	1·0513	1·0332	89	1·0720	1·0455
40	1·0523	1·0340	90	1·0713	1·0447
41	1·0533	1·0348	91	1·0705	1·0438
42	1·0543	1·0355	92	1·0696	1 0428
43	1·0552	1·0363	93	1·0686	1·0416
44	1·0562	1·0370	94	1·0674	1·0403
45	1·0571	1·0377	95	1·0660	1·0388
46	1·0580	1·0384	96	1·0644	1·0370
47	1·0589	1·0391	97	1·0625	1·0350
48	1·0598	1·0397	98	1·0604	1·0327
49	1·0607	1·0404	99	1·0580	1·0301
50	1·0615	1·0410	100	1·0553	1·0273

Vinegar made from dilute alcohol or ripe wines, in which no great excess of albuminous and other matters is present, can to a certain limit be tested with sufficient accuracy by the acetometer; but vinegars made from malt, poor wines, and such liquids as contain an excess of organic matters, do not admit of being tested with the required degree of accuracy by this method, since the apparent quantity of real acetic acid is increased by the presence of foreign bodies, which add to the density of the liquid. In some cases the vinegar is saturated with chalk or milk of lime, the solution filtered, and the specific gravity of the acetate of lime liquor ascertained, by which a nearer approximation is arrived at than by the direct testing of the vinegar; yet on neither of these two methods can implicit reliance be placed.

The most common method of ascertaining the percentage of acetic acid in vinegar is to neutralize it with pure carbonate of potassa or soda, noting the quantity of these salts required to saturate the acid. The amount of real acid in a sample is then ascertained by a simple calculation, since every 106 grains of the pure carbonate of soda, or 138 grains of the carbonate of potassa—one equivalent—indicate 120 grains, or two equivalents, of pure acetic acid. The examination is made in the following manner:—530 grains of pure dry carbonate of soda are dissolved in 10,000 grains of distilled water—ten alkalimetrical measures. This mixture constitutes the test solution, and is kept in a stoppered bottle for use. An ounce of the vinegar is either weighed or measured, and put into a beaker or porcelain dish, and 1000 grains of the test solution—equal to 100 divisions of the burette—are poured, in successive small portions, into the vinegar, the solution being well agitated with a glass or porcelain rod after each addition. The solution is tested at intervals with blue litmus paper, and so long as its colour is reddened when dropped into the liquid, and the effervescing continues, free acid still remains in solution, and more test solution must be added. When the saturation of the vinegar is nearly complete, heat is applied to expel the carbonic acid which is absorbed, and would, if not driven off, communicate a faint rose tint to the test paper, and thus cause an error in the results. If the paper still becomes red after heating, a few drops more of the alkaline liquor are poured in, till the paper is only feebly reddened. Sometimes a few drops of tincture of litmus are added to the acetic acid to be tested; as the operation draws to a close the litmus regains in part its blue colour. This plan, however, offers no advantage, for the acid may be completely saturated long before the colour of the blue litmus is restored. The number of divisions of the test liquor required to saturate the acid is read off, and by a *rule of three* calculation the amount of acid in the sample is found.

Fig. 29 represents Gay-Lussac's burette. Bink's burette, Fig. 30, is much less fragile; the solutions are, however, liable to run down outside the spout, *a*. This can be prevented by applying a little stiff tallow to the spout, and boring through it a small hole with a needle. To manage this instrument properly it must be held near the top, the thumb being placed above the scale. If it be held lower down the delivery of single drops is difficult. The most convenient of all forms of burette is that of MOHR, Fig. 31. *a* is a cylindrical tube which is graduated into 100 parts, 0° being at the top and 100° at the bottom. The tube is open at both ends, but the lower end is contracted, and is connected by a short flexible tube of vulcanized caoutchouc with a small glass jet, *c*. Across the flexible tube is placed a pinch-cock, *d*, which closes the tube when left at rest, and opens it when the buttons at *d* are slightly pressed with the finger and thumb. It is easy either to let out a continuous stream of the test liquor, or to limit its passage to single drops. The burette is suspended by a ring of cork or caoutchouc from the upper arm, *c b*, of the support. The lower arm serves to keep the burette in a vertical position. The point, *c*, is fixed at such a height above the table as to allow free access to the vessel containing the liquid to be tested.

Fig. 29.

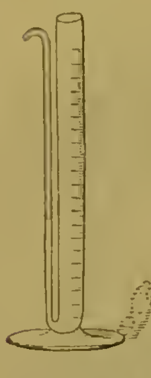


Fig. 30.

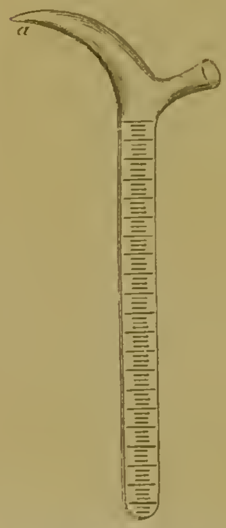
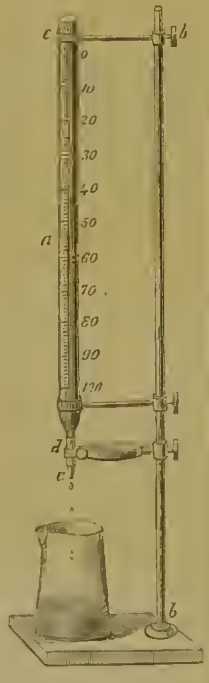


Fig. 31.



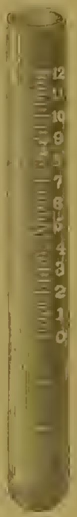
Instead of a soda test liquor, a solution of ammonia is sometimes used to saturate the acid. This solution is prepared by adding water to concentrated ammonia till the spec. grav. is 0·992: 1000 grains of this dilute ammonia contain one equivalent of ammonia, which is capable of saturating one equivalent of acetic acid. The application of this test is similar to that already described.

There is some difficulty in preserving the dilute ammonia of the same strength, which is an objection to its use; but a uniformity of concentration is insured by introducing into the bottle two glass hydrometer bulbs, so adjusted that one remains barely touching at the bottom, and the other floats just under the surface of the liquid, as long as the test liquor maintains the proper strength. If a part of the ammonia volatilizes, the specific gravity of the liquor will become proportionably greater, and the glass bulbs rise; the lower one higher from the bottom, and the upper one partly above the surface. When this happens more strong ammonia is added, till the hydrostatic drops are properly readjusted.

OTTO's acetometer, Fig. 32, is a glass tube sealed at one end, 36 centimètres long by 1·5 centimètres wide; upon it two scales are engraved, one at the bottom for measuring the vinegar, and the scale at the top for measuring the test liquor. His mode of operation is to pour a certain quantity of litmus solution into the tube, and then sufficient vinegar to fill it up to where the upper scale begins. The test liquor, containing 1·369 per cent. of ammonia, is then added until the blue colour of the litmus is restored; the quantity required of course indicates the percentage of acetic acid.

These methods are slightly inaccurate, in consequence of neutral acetates of the alkalies themselves exhibiting an alkaline reaction. The error, however, has been shown by OTTO to be less than 0·1 per cent. for an acid containing 10 per cent. of crystallizable acetic acid. It may, moreover, be avoided by using a test solution which has been graduated for the purpose by means of a solution of pure acetic acid of known strength.

Fig. 32.



GREVILLE WILLIAMS advocates the use of lime saccharate. To prepare this test liquid lime is dissolved in a moderately strong solution of sugar. The solution, after filtering, has its strength ascertained by means of a standard sulphuric acid solution, and is then diluted until five divisions of the burette correspond to one grain of acetic acid. To perform this experiment fifty grains of the sample to be examined are taken and made up with water into about two ounces of liquid, a few pieces of litmus paper are then thrown in and the test liquid carefully added, with constant stirring, until the blue colour of the litmus is restored. With moderate care the results do not vary, and in rapid work can be relied on to 0·25 per cent.

In each of the preceding modes of testing it is evident that, should the vinegar contain any admixture of other acids, such as sulphuric acid, hydrochloric acid, or the like, these will increase the quantity of the alkaline solution required to neutralize the weight of vinegar taken for the test. Preliminary examinations should be made to ascertain if these impurities be present, and if so, their amount determined.

SULPHURIC ACID IN VINEGAR.—After the conversion of malt, beer, weak wines, &c., into vinegar, a putrid fermentation often takes place which decomposes the whole of their acetic acid. It was formerly considered necessary that sulphuric acid should be added to counteract this tendency of the liquid to decomposition, and to preserve it from turbidity. This addition was permitted to the extent of 1 gallon of sulphuric acid to 1000 gallons of vinegar, by an excise regulation, and had therefore a legal sanction. Sulphuric acid is now known to be unnecessary in properly prepared vinegars, although it is still added by some manufacturers for the purpose of increasing the strength of their vinegars, or in some instances merely from habit and the indisposition to disturb the routine of an old-established practice. Sulphuric acid in vinegar should invariably be looked upon as the mark of inferior quality; for it is only where the mode of manufacture is defective that its addition is at all necessary. Sulphuric acid does not in the least favour digestion, and it is known that it affects the coats of the stomach. Besides the addition of the $\frac{1}{1000}$ part of sulphuric acid, many persons—makers or vendors—add a far greater proportion in order to confer that acidity which ought only to be produced by the acetic acid. Some of these mixtures are most dangerous to the community, as the commonest oil of vitriol is often used as the adulterant. Vinegars adulterated with pyrites vitriol also generally contain arsenic.

Several methods have been given by chemists for the detection of sulphuric acid in vinegar or acetic acid. Here will be enumerated a few of the more trustworthy for the qualitative and quantitative estimation of sulphuric acid in vinegars. It should, however, be borne in mind that vinegar made from wine, malt, &c., contains some soluble neutral sulphates, these being natural constituents of the grain or grapes from which the wine or fermented wort is obtained; and that by the more delicate tests this quantity of sulphuric acid in combination will be indicated.

If the vinegar be suspected to contain a greater quantity of sulphuric acid than that which is authorized, it may be ascertained pretty accurately by making a solution of 1 part of sugar in 30 parts of water, bringing the liquid to a temperature of 190° or 200° Fahr. by means of steam heat, and then adding a drop of the suspected vinegar. If sulphuric acid in excess be present it will carbonize the sugar, causing a blackish spot to appear at the point where the vinegar came in contact with the saccharine solution. This happens when the vinegar contains the $\frac{1}{300}$th of its weight of the adulterant. When the amount ranges between the $\frac{1}{600}$th and $\frac{1}{800}$th of the impurity, a greenish spot appears.

The next and principal test is the precipitation of the sulphuric acid by means of a soluble salt of barium, in the form of insoluble barium sulphate ($BaSO_4$), which falls down as a heavy white powder. A correct inference may be drawn as to whether the vinegar contains more than the ordinary amount of sulphuric acid, by observing the bulk of the precipitate. An immediate white precipitate indicates the presence of sulphuric acid, when only about the $\frac{1}{200}$th or $\frac{1}{1000}$th of the adulterant is present; but this precipitate does not so quickly subside as when larger quantities are present.

Sulphuric acid is determined quantitatively in the following way:—Half a pound of the vinegar is evaporated in a porcelain or platinum basin, on a water-bath or by steam heat, till the eight ounces are reduced to one ounce; the basin is then removed from the heat and allowed to cool, and five to six times its bulk of strong alcohol is added. The earthy and alkaline sulphates naturally present in the vinegar are insoluble in alcohol, and consequently fall down. After the precipitate has subsided, the solution is filtered off, and the residue washed with dilute spirit. The greater part of the alcohol is then expelled by evaporation in the water-bath, the remaining liquid diluted with ten or twelve times its volume of water, and a solution of chloride of barium poured in as long as a precipitate is formed. The precipitate, after subsidence and filtration of the supernatant liquid, is digested with moderately dilute warm hydrochloric acid, then thrown upon a filter, washed with boiling water till a portion of the washings gives no white precipitate upon addition of sulphuric acid, dried in a water or air bath, and when dry, burned in a platinum crucible. The precipitate should be detached as much as possible from the filter-paper, and the latter burned in the crucible until the whole of the carbonaceous matter of the paper is destroyed, and the precipitate then introduced, heated to redness, and weighed. Every 233 parts of this precipitate ($BaSO_4$) indicate 98 parts of sulphuric acid (H_2SO_4).

When great accuracy is not required, a standard solution of barium chloride is used: 500 grains of vinegar, diluted with twice its weight of water, are poured into a tall and not very wide beaker glass, or precipitating jar, and the graduated burette is filled, and the standard solution poured gradually into the vinegar, in small portions at a time, the mixture being well stirred with a glass rod after each addition; the whole left to rest in a warm situation till the precipitate has collected at the bottom, after which another addition of the test solution is made, observing the same rule as above given, so long as any precipitate forms. When the last drop causes no precipitate, or at least only a very slight one, the number of measures of test solution added are read off, and from this the amount of sulphuric acid is calculated.

Some spring and river waters contain a large quantity of sulphates, in which case a greater amount of the test solution will be required than for the free sulphuric acid in the vinegar; an examination of the water employed should therefore precede the testing of the vinegar, or the analysis should be conducted by the first method described, namely, evaporation of the vinegar, and treatment with alcohol before precipitation.

HYDROCHLORIC ACID IN VINEGAR.—This acid is not frequently met with in vinegar. Its presence may be ascertained by distilling a quantity of the vinegar from a glass retort, to which a condenser is attached, and adding a few drops of a solution of silver nitrate to the distillate. A white precipitate indicates the presence of hydrochloric acid. To determine quantitatively the amount of the acid, 8 or 16 ozs. of the vinegar are distilled, till the whole of the liquid has passed over into the receiver. A solution of silver nitrate is poured into the distillate as long as a precipitate occurs. The solution is left at rest till the white curdy precipitate of silver chloride falls to the bottom, which is filtered off, washed with a little dilute nitric acid first, and afterwards with distilled water; dried at 212° Fahr. (100° C.) in a water-bath, ignited in a porcelain crucible, and weighed. From the weight of the silver chloride, that of the hydrochloric acid is calculated.

Nitric Acid in Vinegar.—This acid is rarely employed to adulterate vinegar. If its presence be suspected, about 8 ozs. are neutralized with sodium carbonate, the liquid evaporated to dryness, and the residue distilled with a few drops of strong sulphuric acid. The distillate is received in an ice-cold receiver, neutralized with potassa, and a solution of starch paste and potassium iodide added. Iodine forms a deep indigo blue compound with starch, which is soluble in pure water, but insoluble in solutions containing free acid; if a blue colour appears, it is an infallible proof of the presence of nitric acid. A simpler test is to boil a portion of the residue of the vinegar after evaporation with hydrochloric acid and copper turnings; if nitric acid be present, red fumes of nitrous acid, possessing a very characteristic vapour, are evolved.

Tartaric Acid in Vinegar.—On evaporating a portion of the vinegar in a water-bath, if tartaric acid be present, a viscid mass of the consistence of treacle, and highly acid, remains. By adding alcohol to this substance, and agitating for a short time, the tartaric acid is dissolved; the spirituous extract is then filtered off, mixed with potassium chloride, and well agitated, upon which it yields a crystalline precipitate of potassium bitartrate ($KC_4H_5O_6$)—cream of tartar—in the presence of tartaric acid. It must be remarked that wine vinegar naturally contains some tartaric acid in the form of cream of tartar—this compound being one of the solid constituents of the grapes.

Metallic Salts in Vinegar.—The salts which are formed in vinegar arise from the action of the acid on the metallic vessels employed. Vinegar made by the *quick* process, or in *vinegar fields*, never contains any of these compounds, unless the vinegar is afterwards distilled in metallic vessels. At a high temperature, with access of air, acetic acid acts on the metallic parts of the still or condensing worm. As copper, lead, tin, and zinc, are generally the materials used in the construction of the still or worm, these are the only bodies that have to be looked for in the vinegar. A portion of the vinegar is submitted to a stream of sulphuretted hydrogen gas; if a black colour or precipitate be produced, copper or lead is present. Another portion (about 10 ozs.) of the vinegar is evaporated to dryness in a basin, and the residue heated to redness in a porcelain crucible, and the whitish ash remaining treated with a few drops of nitric acid, heated, and filtered; if, on treating the solution with ammonia, a more or less blue colour is given to the liquid, copper is present. Tin gives a yellow precipitate with sulphuretted hydrogen. If, on the addition of ammonia, or ammonium sulphide, a white precipitate is formed, zinc is present; if the precipitate is black, or has a greyish cast, there is likewise a small proportion of iron. The quantity of salts of these metals found in vinegar is generally so very small as to escape detection, unless a large quantity of the vinegar is evaporated, and the residue submitted to a thorough chemical examination.

Should pepper, chillies, &c., be added to vinegar for the purpose of conferring more pungency, they may be detected by neutralizing the acid with carbonate of soda, and tasting the liquor; if these bodies be present the solution will still retain the sharpness peculiar to such spices.

Flies (*musca cellaris*) and eels (*vibrio aceti*) are often found in vinegar; they may be destroyed by passing the vinegar through tubes immersed in boiling water.

The average percentage of fixed matter which remains after evaporating the different vinegars to dryness is as follows:

Wine Vinegar,	2·05 to 2·10
Beer Vinegar,	5·00 to 6·00
Cider Vinegar,	1·40 to 1·50

Vinegar is sometimes concentrated by exposing it to cold, and separating the layers of ice; although the greater part of the water is by this treatment removed, yet a large quantity of acetic acid is likewise abstracted, and where the vinegar is very dilute the process is not at all economical. Another method is, to keep the vinegar heated at a temperature between 212° and 220° Fahr. (100° to 104° C.); for while water boils at 212°, the hydrated acetic acid boils at 248° Fahr. (120° C.), so that at 212° a large proportion of water is driven off and very little acid; every disadvantage is removed when the boiling point of the vinegar is elevated. Stein recommends for this purpose the addition of common salt, in the proportion of 30 lbs. for every 100 lbs. of vinegar. The acid then distils over without loss, and is obtained much stronger than in the ordinary mode of distillation.

Some cautions may here be given with reference to the vessels in which vinegar is kept, as these often consist of metals. A painted vessel should never be used; the basis of nearly all pigments is white lead, which readily dissolves in acetic acid, forming sugar of lead—lead acetate; and many paints contain copper and other metallic salts, soluble in acetic acid, any of which may prove poisonous. Iron is easily attacked by acetic acid; and although the salt which is formed in the liquid—the iron acetate—is not injurious in small quantity, yet it communicates to food a disagreeable taste.

Common earthen vessels, glazed with oxide of lead—litharge, red lead—part of which is frequently uncombined with the body of the ware, should not be employed; for the acid would undoubtedly dissolve that portion of the oxide, and might even attack the well-burned porcelain itself, the more so if the acid be heated in the vessel. Salt-glazed stoneware, good English pottery, porcelain, glazed or enamelled iron, silver, and copper (if kept quite bright), may all be employed for boiling acetic acid or its preparations with safety.

The proof vinegar of the excise has a spec. grav. of 1·0085, and contains about 5 per cent. of acetic acid. In commerce this vinegar is represented by No. 24, from the fact that 24 grains of pure dry carbonate of soda are required to neutralize a fluid ounce. Weaker vinegars are represented by the Nos. 18, 20, 22, according to their strength; and, as in the foregoing instance, these figures equal the number of grains of carbonate of soda that will saturate a fluid ounce.

Acetic acid is extensively employed to preserve animal and vegetable substances from decay or putrefaction. Wood vinegar is preferable to other varieties, on account of essential oils which it contains in small quantities, and which render its antiseptic properties more active. The creasote of this acid confers a smoky taste on meats.

Acetic acid is also employed to a large extent in the arts, chiefly in calico-printing and dyeing. When required for these uses it is united to bases, with which it forms acetates, principally used as mordants. It is likewise used in the preparation of varnishes, for dissolving gums and albuminous bodies. Aromatic vinegar is used medicinally.

ALUMINIUM TRIACETATE. — *Neutral Acetate of Alumina, Sesquiacetate of Alumina, Red Liquor.* $Al_2^{vi}(C_2H_3O_2)_6$.—This salt has never been obtained in the dry state. It can be prepared by decomposing a concentrated solution of aluminium sulphate by a concentrated solution of lead acetate:—

$$Al_2^{vi}(SO_4)_3 + 3Pb(C_2H_3O_2)_2 = 3PbSO_4 + Al_2^{vi}(C_2H_3O_2)_6.$$

It is likewise obtained when recently precipitated aluminium hydrate is digested with strong acetic acid.

WALTER CRUM considers that no true triacetate exists, but that the solution contains aluminium diacetate with an equivalent of free acetic acid. In support of this view, he proves that when means are taken to evaporate the aluminous solution at a low temperature with sufficient rapidity a dry substance is obtained, which may be redissolved easily and entirely by water. This is the diacetate:—

$$Al_2^{vi}\left\{\begin{matrix}(C_2H_3O_2)_4\\ O\end{matrix}\right. + 4H_2O.$$

If the triacetate solution is allowed to stand for a few days in the cold a white precipitate is formed, which contains five equivalents of water:—

$$Al_2^{vi}\left\{\begin{matrix}(C_2H_3O_2)_4\\ O\end{matrix}\right. + 5H_2O.$$

This modification is insoluble in water, slightly soluble in weak acids, and very soluble in caustic alkalies.

If the solution of the neutral acetate is heated to 212° Fahr. (100° C.) another basic acetate is obtained, containing two equivalents of water and having the composition—

$$Al_2^{vi}\left\{\begin{matrix}(C_2H_3O_2)_4\\ O\end{matrix}\right. + 2H_2O,$$

at the boiling temperature; the liquid is thus deprived in about half an hour of the whole of its alumina, which goes down with two-thirds of the acetic acid, leaving one-third in the liquid.

Soluble Diacetate with four Equivalents of Water.—Notwithstanding the tendency of a concentrated solution of aluminium triacetate to deposit the insoluble salt, it may be evaporated, with certain precautions, to a dry substance soluble in water. For this purpose it must be spread very thin over sheets of glass or of porcelain, and exposed to a heat not exceeding 100° Fahr. (38° C.); and as it runs together into drops, like water upon an oiled surface, it must be constantly rubbed with a platinum or silver spatula. If these precautions are neglected, a mixture is obtained of the insoluble with the soluble acetate. The soluble salt is thus produced in scales having the appearance of gum when moistened, and leaving no residue when dissolved in water.

Aluminium diacetate may be produced at once in solution, and is the most suitable combination from which to form dry soluble diacetate. CRUM'S mode of proceeding is as follows:—Dissolve 24 parts of precipitated aluminium diacetate in 15 of rectified sulphuric acid, and 40 of water. Dilute further with 100 parts of water, and add about 44 parts of lead carbonate to precipitate the sulphuric acid. Filter the solution, and pass sulphuretted hydrogen through it until it ceases to precipitate lead, and then add barium acetate so long as it is precipitated by the sulphuric acid of the lead sulphate which had remained in solution. When in this state, if the mixture be well agitated for half an hour in an open vessel, the excess of sulphuretted hydrogen will be removed, and it may be filtered without the danger of the filtrate becoming afterwards milky. A solution is thus obtained containing about 4 per cent. of aluminium diacetate, which is the strongest solution even of this modification which can be preserved for any length of time without depositing an insoluble salt. Spread thinly over sheets of glass it evaporates at 60° to 100° Fahr. (15° to 38° C.) without running into drops as does the triacetate solution. The scales which it forms are transparent and soluble in water. The solution of diacetate has no smell of acetic acid at ordinary temperatures. Its formula is—

$$Al_2^{vi}\left\{\begin{matrix}(C_2H_3O_2)_4\\ O\end{matrix}\right. + 4H_2O;$$

or in equivalents—

$$(Al_2O_32\overline{A} + 4Aq).$$

Insoluble Diacetate with five Equivalents of Water.—Triacetate solution is produced by mixing together strong solutions of aluminium sulphate and lead acetate (as already described). The solutions are poured slowly together in a vessel surrounded with cold water, and much agitated to reduce the temperature, and filtered when cold. Any lead remaining in solution is precipitated by sulphuretted hydrogen, sulphuric acid by barium acetate. The strongest solution formed in this way contains about 5 per cent. of alumina (from about 6 lbs. of lead acetate in an imperial gallon of the mixture). This solution is left at rest for four or five days, when it begins, without losing much of its transparency, to deposit a crust which continues to increase in thickness. When the liquid is poured off and the crust allowed to dry, it separates readily from the vessel in hard plates like porcelain. If the solution be not left strictly at rest, it becomes turbid after some days, and the crust is produced in a more friable state. In cold weather this solution remains unaltered for a much longer time. Heated to redness, the salt becomes black from the decomposition of part of its acetic acid, and in this state it very slightly affects the colour of moistened litmus paper. Its analysis agrees with the formula—

$$\mathrm{Al}_2^{vi}\left\{\begin{matrix}(\mathrm{C_2H_3O_2})_4 \\ \mathrm{O}\end{matrix}\right. + 5\mathrm{H_2O}\ ;$$

or in equivalents—

$$(\mathrm{Al_2O_3}2\overline{\mathrm{A}} + 5\ \mathrm{Aq}).$$

Insoluble Diacetate with two Equivalents of Water.—If heat be applied to the strong solution of aluminium triacetate described above, it speedily becomes turbid and deposits a white heavy powder, which falls readily to the bottom of the vessel. In a couple of days at 100° Fahr. (38° C.), a considerable quantity of this powder is deposited; but in two or three hours at 160° Fahr. (71° C.), and in a much shorter time at boiling heat, the whole of the salt is thrown down, and nothing remains in the liquid but acetic acid and a trace of alumina. The precipitate has a crystalline shining appearance in the moist state, and seems under the microscope to consist of small oval particles of uniform size. It falls into fine powder on drying, after which when mixed with water it remains a long time in suspension.

When heat is applied to a solution of triacetate containing only 3 per cent. of alumina it yields the insoluble diacetate; but in that case not acetic acid only, but a considerable quantity of aluminium acetate also, remains in solution. Solutions containing 2 per cent. of alumina are precipitated by boiling, if they have been kept for some weeks, but not if recently prepared.

In whatever way deposited this substance is exceedingly insoluble in acetic acid. It dissolves in sulphuric, hydrochloric, or nitric acid, forming aluminium salts and liberating acetic acid. Analysis gives results agreeing with the formula—

$$\mathrm{Al}_2^{vi}\left\{\begin{matrix}(\mathrm{C_2H_3O_2})_4 \\ \mathrm{O}\end{matrix}\right. + 2\ \mathrm{H_2O}$$

Red Liquor.—A solution of aluminium triacetate (or diacetate, plus one equivalent of acetic acid) extensively used as a mordant liquor by calico-printers. So highly esteemed is aluminium acetate for forming dense opaque lakes, that one-fourth of the total quantity of alum manufactured in this country is utilized in this manner.

Sesquiacetate of alumina, as it is termed commercially—technically *red liquor*, from its being coloured with lichens—is always met with in the liquid state. It is manufactured for the use of calico-printers by adding to every gallon of calcium acetate liquor $2\frac{3}{4}$ lbs. of alum, agitating the mixture briskly, and then leaving it to rest, in order that the calcium sulphate may settle down. The decomposition of the calcium acetate is known by testing a small portion of the filtered liquid in a tube with a concentrated solution of alum; if a precipitate of calcium sulphate falls more alum must be added, till the acetate of lime is completely decomposed. The liquor is next filtered off, and the solution concentrated by evaporation till it acquires a spec. grav. of 1·087 to 1·100; allowed to repose for some time to deposit any sulphate of lime, and then drawn off for use or for market. The quality of this liquid as a mordant is inferior to that next described, on account of the imperfect decomposition of the lime salt, and the presence of a small portion of lime still retained in the red liquor, which impairs very much the beauty and gloss of the colour given to the cloth.

A better mordant is made by decomposing alum by lead acetate. Since lead sulphate is insoluble, the decomposition of the alum solution is more perfect than when it is acted upon by acetate of lime; nevertheless, *red liquor* is not a true acetate, but a mixture of aluminium acetate, sulphate, and hydrate, with potassium sulphate, as will be seen from the recipes in general use for its manufacture. In practice, in place of using quantities equivalent to the production of aluminium triacetates, it is found advantageous to employ equal parts of alum and sugar of lead, or even a rather less quantity of the latter. The alum is dissolved in boiling water, and the powdered lead acetate added to the solution. About one-tenth of crystallized carbonate of soda, or a little carbonate of lime, is added to the alum, to combine with the free acid. The three following recipes serve to indicate the proportions employed:—

No. 1.—Dissolve 100 lbs. of alum in 50 galls. of boiling water, and add 100 lbs. of acetate of lead in fine powder, stirring the mixture well at first, and likewise several times during the cooling.

The clear supernatant liquid consists of an aluminium diacetate, potassium sulphate, and a little basic aluminic sulphate.

No. 2.—Dissolve 100 lbs. of alum in 50 galls. of water; to the solution add slowly 10 lbs. of crystallized carbonate of soda, and then stir in 100 lbs. of acetate of lead in powder.

No. 3.—Dissolve 100 lbs. of alum in 50 galls. of boiling water, and add in small portions 6 lbs. of crystallized carbonate of soda, and then stir in 50 lbs. of acetate of lead in powder, as before.

Nos. 2 and 3 contain acid aluminium diacetate, basic aluminic sulphate dissolved in acetic acid, and potassium and sodium sulphates. No. 2 becomes cloudy at 154° Fahr., and gelatinizes at 165° (68° and 74° C.);

No. 3 clouds at 175°, and gelatinizes at 192° (80°, 89° C.); the cloudiness disappears on cooling, and is entirely prevented by an excess of alumina in the solutions. According to the experiments of KÖCHLIN SCHONCH, it appears that the activity of the red liquor is not wholly dependent upon the amount of aluminium acetate which it contains, since a portion of the salt is converted into a basic sulphate which combines with the triacetate and constitutes the true mordant.

When used by the calico-printer the red liquor is thickened with gum or some other suitable material, and with it the design is impressed upon the cloth by a wood block, or by any other means; on subsequently submitting the goods to the drying-bath, acetic acid is partly volatilized, and the aluminous basic compound remains perfectly combined with the cloth.

It is immaterial, as to the effect on the texture, and the beauty of colour produced on the cloth, whether 100 lbs. of alum be decomposed by 125 or 75 lbs. of acetate of lead, since the aluminium acetate acts by giving up its base to the fibre of the cloth, and this is effected as well when a basic sulphate of the earth is present as when it is wholly in the form of a sesquiacetate. RUNGE differs from this view; by his experiments he endeavours to show that the quantity of acetate of lead should be always 120 lbs. to every 100 lbs. of alum, and that even the amount of water employed affects the quality of the product. He based equal weights of cotton fabric in each of the following solutions, made by adding—

100 lbs. of alum,
75 lbs. of acetate of lead, and
280 lbs. of water together, agitating the mixture, and filtering:

Secondly, by dissolving—

100 lbs. of alum in
448 lbs. of water, and adding
120 lbs. of acetate of lead in powder, agitating the mixture, and filtering off the clear liquor as above.

The fabrics being allowed to remain in an equal measure of these solutions for the same time, and dried at the same temperature, were washed with equal quantities of hot water; the washings from the cloth mordanted in the first solution contained much alumina, while only very slight traces were indicated by the washings from the cloth steeped in the second solution. He did not, however, take into account the neutralization of a part of the sulphuric acid by carbonate of soda, which, by bringing basic salts into action, materially changes the reaction which takes place during drying, and undoubtedly fixes a much larger amount of alumina in the fabric than would otherwise be the case. And although a portion of this alumina may be washed out after drying, it is questionable whether the advantage of using so large a proportion of lead acetate is not counterbalanced by the greatly increased cost. The usual addition of carbonate of soda, to the extent of about one-tenth of the alum employed, acts advantageously in the manufacture of red liquor, when a low proportion of sugar of lead is used, by uniting partly with the sulphuric acid, and producing a basic sulphate, as well as acetate of alumina, as mentioned above.

CRACE CALVERT states, from practical observations, that a sulphacetate of alumina is to be preferred, as giving the most satisfactory results. He considers that a mordant of such a composition is the best adapted for fixing the colours, on account of the excess of alumina in such a solution, above those which contain, besides the aluminous salts, salts of the alkalies, which are inert in the uses for which red liquor is manufactured.

The preceding he prepares by mixing together—

453 lbs. of ammonia alum,
379 lbs. of lead acetate, and
1132 lbs. of water;

or,

383 lbs. of aluminium sulphate,
379 lbs. of lead acetate, and
1132 lbs. of water;

or,

453 lbs. of alum, and a quantity of solution of acetate of lime, amounting to 158 lbs.;

or,

333 lbs. of aluminium sulphate, with the same amount of acetate of lime solution.

On agitating the foregoing mixtures, decomposition takes place; sulphate of lead or of lime is thrown down, and a sulphacetate remains, with an equivalent of ammonium sulphate from the ammonia alum. Instead of alum, many printers now use aluminium sulphate—

$$Al_2^{vi}(SO_4)_3 + 18H_2O;$$

or,

$$Al_2O_33SO_3 + 18H_2O;$$

in the fabrication of this mordant, which is much more economical, as the solution of this salt, brought to the standard strength, or 1·085 spec. grav., contains more alumina than the ordinary red liquor of that strength, as the following analyses show:—

	Formula. $Al_2^{vi}SO_4,4C_2H_3O_2 + (NH_4)_2SO_4$.		Formula. $Al_2^{vi}SO_4,2C_2H_3O_2 + (NH_4)_2SO_4$.	Formula. $Al_2^{vi}SO_4,4C_2H_3O_2$.
	Mordant A.	Mordant B.	Mordant C.	Mordant D.
	Grs. Oz. Grs.	Grs. Oz. Grs.	Grs. Oz. Grs.	Grs. Oz. Grs.
Alumina,............................	1680·0 = 3 368	1830·0 = 4 80	1239·0 = 2 365	2164·4 = 4 414
Acetic acid,............................	3369·8 = 7 307	3570·0 = 8 170	1281·7 = 2 406	3679·2 = 8 179
Sulphuric acid,............................	2642·5 = 6 17	2800 0 = 6 175	3017·0 = 6 392	1664·6 = 3 352
Ammonia and water,............................	674·1 = 1 236	910·0 = 2 35	653·1 = 1 215	

In 1872 Messrs. STORCK & Co., of Asnières, France, patented a process for the manufacture of aluminium acetate from the phosphate. Aluminium phosphate is converted into acid phosphate by dissolving it in phosphoric acid. To this liquor sufficient lead acetate is added to precipitate the whole of the phosphoric acid. Soluble aluminium acetate and insoluble lead sulphate are thus formed. The aluminium acetate is then separated by filtration, and subsequently treated in a similar manner to that obtained for industrial purposes by the double decomposition with aluminium sulphate. The phosphate of lead is either used to produce pure phosphoric acid by decomposing it by sulphuric acid or sulphuretted hydrogen, or an alkaline phosphate is formed thereof by treating it with an alkaline sulphide. It may likewise be used for the production of phosphorus; in this case it is mixed with charcoal and subjected to distillation.

Ammonium Acetate, *Liquor Ammoniæ Acetatis* or *Spiritus Mindereri*, Pharmacopœia; *Neutral Acetate of Ammonia*, $NH_4,C_2H_3O_2$.—This substance is a white inodorous salt. It is obtained by saturating glacial acetic acid with dry ammonia gas. It is very difficult to obtain in the crystalline form, on account of its aqueous solution giving off ammonia when evaporated, thus becoming converted into the acid salt. When heated it evolves first ammonia, then acetic acid, and lastly acetamide (C_2H_5NO). It melts at 192° Fahr. (89° C.); inclosed over oil of vitriol it loses 9 per cent. of its weight and becomes transformed into the acid salt.

In medicine it has long been used as a diaphoretic.

It was formerly obtained by saturating distilled vinegar with carbonate of ammonia. The method now adopted is to reduce carbonate of ammonia to powder, and add it gradually to acetic acid until a neutral solution is formed. The proportions used are—acetic acid, 10 fluid ounces; ammonium carbonate, 3½ ounces (or a sufficiency); then add 2½ pints of distilled water.

Ammonium Acid Acetate, Diacetate, $NH_4C_2H_3O_2,C_2H_4O_2$.—When equal weights of ammonium chloride and potassium acetate are distilled together at a moderate temperature, ammonia is at first eliminated; and afterwards ammonium diacetate distils over in the form of an oily liquid, which concretes into acicular crystals, which are deliquescent, and are dissolved in all proportions by water and alcohol.

$$\overset{\text{Ammonium chloride.}}{2NH_4Cl} + \overset{\text{Potassium acetate.}}{2KC_2H_3O_2} = \overset{\text{Ammonia.}}{NH_4O} + \overset{\text{Ammonium diacetate.}}{NH_4,C_2H_3O_2,C_2H_4O_2} + \overset{\text{Potassium chloride.}}{2KCl.}$$

Dry ammonia, transmitted into the fused diacetate, converts it into the solid neutral acetate—

$$\overset{\text{Diacetate.}}{NH_4C_2H_3O_2,C_2H_4O_2} + \overset{\text{Ammonia gas.}}{NH_3} = \overset{\text{Neutral acetate,}}{2NH_4C_2H_3O_2.}$$

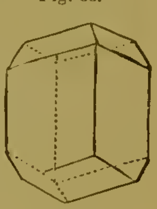
Diacetate of ammonia forms striated prisms, fusible at 168° Fahr. (76° C.), and subliming unchanged at 248° Fahr. (121° C.).

Acetates of Copper. *Cuprous Acetate*, $Cu_2(C_2H_3O_2)_2$.—This salt is produced by the action of heat upon cupric acetate (verdigris, described below). It is a white substance crystallizing in fine needles; it is decomposed by water into cupric acetate and yellow cuprous hydrate. It reddens litmus, and has a caustic astringent taste. According to Berzelius this body is contained in common green verdigris, and sublimes when that substance is distilled.

Normal Cupric Acetate, Crystallized Verdigris; Verdet, Cristaux de Venus, French; $Cu(C_2H_3O_2)_2$.—The normal acetate of copper is formed by dissolving the cupric oxide, or common verdigris, in acetic acid, or by precipitating a solution of sulphate of copper by normal acetate of lead or baryta, filtering off the precipitated sulphate of lead or baryta, and evaporating the filtered liquid, and crystallizing. In each case the liquor must be highly concentrated and left in a cool place.

The crystals are dark bluish prisms (Fig. 33), belonging to the monoclinal system, and containing $Cu(C_2H_3O_2)_2 + H_2O$. This salt is efflorescent, soluble in about 13½ parts of cold, and 5 of boiling water, possesses a disagreeable metallic taste, and is poisonous like all soluble copper salts. The crystals, after drying in vacuo, lose no more water at 212° Fahr. (100° C.), but give off 9 per cent. of their water between 230° Fahr. (110° C.), and 284° Fahr. (140° C.). Between 496° Fahr. (240° C.) and 532° Fahr. (260° C.) glacial acetic acid is given off. When heated strongly in the air crystals of verdigris take fire and burn with a vivid green flame.

Fig. 33.



Acetate of copper was formerly employed in the manufacture of acetic acid. Cuprous oxide (Cu_2O) is obtained in red octahedral crystals when the neutral salt is heated with organic substances, such as sugar, honey, starch, &c.

When commercial verdigris is dissolved in dilute acetic acid, and the salt crystallized at 40° to 45° Fahr. (5° to 8° C.), an acetate with five atoms of water is obtained, in beautiful blue oblique four-sided prisms.

$$Cu(C_2H_3O_2)_2 + 5H_2O.$$

On raising the temperature to about 86° Fahr. (30° C.), the crystals almost instantaneously lose their blue colour, and acquire a greenish hue; four atoms of water are expelled, and neutral acetate remains, with one atom of water.

Its chief use in the arts is in making pigments, and for resisting the blue colour which the indigo would communicate in the indigo-bath of the calico-printer. In the latter case, its mode of action depends on the readiness with which it parts with oxygen, whereby the indigo is oxidized before it can exert any action on the cloth, being itself reduced to the state of acetate of suboxide of copper. Crystallized verdigris is occasionally employed as a transparent green water-colour or wash for tinting maps. In medicine it is used for external application.

Verdigris.—*Ærugo; vert-de-gris*, French; *grün span*, German. A mixture of the basic cupric acetates. These are three in number:—

Sesquibasic, ...	$(Cu(C_2H_3O_2)_2)_2CuO + 6H_2O$
Dibasic, ...	$Cu(C_2H_3O_2)_2CuO + 6H_2O$
Tribasic, ...	$Cu(C_2H_3O_2)_2 2CuO + 3H_2O$

Sesquibasic cupric acetate is found in all the verdigris of commerce. It is prepared by lixiviating common verdigris with tepid water, and leaving the filtrate to evaporate spontaneously. It is obtained more nearly pure by adding ammonia, little by little, to a boiling concentrated solution of the normal acetate, until the precipitate which is at first formed is redissolved. The new salt then crystallizes out as the liquor cools. The crystals are in pale blue scales; at 212° Fahr. (100° C.) they lose 10·8 per cent. of their water. Their aqueous solution is decomposed by boiling, acetic acid being given off, and the black oxide of copper precipitated.

According to BERZELIUS, green verdigris is a mixture of this salt with small quantities of all the other acetates of copper.

Dibasic cupric acetate constitutes the greater part of the blue variety of verdigris manufactured at Montpellier and other places in the south of France. It forms beautiful, delicate, silky, blue, crystalline needles and scales, which, when ground, form a fine blue powder. When heated to 140° Fahr. (60° C.) they lose 23·45 per cent. of water, and become transformed into a beautiful green, a mixture composed of the neutral and tribasic acetates.

$$\overset{\text{Dibasic Cupric Acetate.}}{2Cu(C_2H_3O_2)_2CuO} = \overset{\text{Normal Acetate}}{Cu(C_2H_3O_2)_2} + \overset{\text{Tribasic Cupric Acetate.}}{Cu(C_2H_3O_2)_2 2CuO.}$$

By repeated exhaustion with water the dibasic is resolved into the insoluble tribasic salt, and a solution of the normal and sesquibasic cupric acetates.

Tribasic cupric acetate is the most stable of any of the acetates of copper. It is prepared by boiling the aqueous solution of the neutral acetate, by heating it with alcohol, by digesting its aqueous solution with cupric hydrate, or by exhausting blue verdigris with water, as just mentioned. The first methods yield the salt in the form of a bluish powder composed of needles and scales; the last as a bright green powder. This salt gives off all its water at 352° Fahr. (160° C.); at a higher temperature it decomposes and evolves acetic acid. Boiling water decomposes the solid tribasic acetate into a brown mixture of the same salt with cupric oxide.

Blue Verdigris.—This salt is formed for commercial purposes by exposing thin copper plates in a confined space, to the combined action of air and moisture, or by submitting copper plates to the action of fermenting refuse from the wine factories (technically *marcs*); in the course of a few days a coating, consisting for the most part of dibasic cupric acetate, forms on the plates, which may be scraped off, and the remaining part of the plate submitted to a fresh operation, till all the copper is converted into verdigris.

The manufacture of verdigris on the large scale is conducted as follows:—In France the chief seats of this manufacture are at Grenoble and Montpellier, where the operations are conducted in a rude but effective way, and usually by women. Husks and refuse of grapes from the wine factories, not entirely exhausted of their juice, are spread loosely in casks until the acetous fermentation takes place. The casks or vessels are covered with matting to protect them from dirt. The limit to which fermentation of the "mares" should be carried, is known by introducing a test sheet of copper into the mass for twenty-four hours; if, on withdrawing it at the end of that time, it is found covered with a uniform green coating, the proper degree of fermentation is reached, otherwise the mass is allowed to remain a day or two longer. Alternate layers of sheets of copper of $\frac{1}{24}$th of an inch thick, and the fermented mares are introduced into large casks, observing that the top and bottom layers are of the latter.

Sheets of copper are prepared by hammering bars of the metal to the above thickness—the more compact the copper sheets the better—and they are then cut out into pieces of 6 or 8 inches long by 3 to 4 broad. These plates are immersed in a concentrated solution of the verdigris, and dried over a charcoal fire; then heated to about 200° Fahr., being held by a cloth in the hand, and packed in the vessels with layers of the fermented husks, as above-mentioned. If the plates be not immersed in the solution of the acetate before packing in the casks with the fermented stalks and skins of the grape, they are liable to be covered with a black coating instead of the green acetate. The quantity of metal required to fill each vessel is between 30 and 40 lbs. Twenty days are sufficient to complete the corrosion of the copper sheets, and induce their combination with the acetic acid present in the mares, but often sixteen or twelve days perfect the work. After this period the upper layer of mares will appear whitish, and if the whole has worked favourably the plates will be covered with silky crystals of a green colour. The plates are then taken from the casks, and dried in the air for two or three days, after which they are moistened with water and again placed to dry, by laying them upright against each other for a week. This process of moistening with water is continued at regular intervals of a week for six or eight times. By this mode of operation the plates swell and become encrusted with increasing coatings of the copper salt, which are detached from the remainder of the plates by a copper knife. The scraped plates are submitted to a fresh treatment, till the whole of the copper is converted into verdigris. After scraping off the salt it is made into a thick consistent mass by kneading it with a little water, and in this state it is packed into leathern bags, which are placed in the sun to dry, until the mass hardens and forms the tough substance which constitutes the commercial article.

Green Verdigris.—At Grenoble the process is nearly the same as above, excepting that, instead of moistening the plates with acetous liquors, acetic acid is employed. In Germany, Sweden, and England, woollen cloths steeped in pyroligneous acid are used, which are placed alternately with the copper plates in a square wooden box. The cloths are moistened with pyroligneous acid every three days for twelve or fifteen days, when small crystals begin to form on the plates. So soon as this happens the cloths are partly withdrawn, and a space allowed for the circulation of the air, the whole being moistened weekly with water. Generally five or six weeks elapse before the completion of the work.

Cupric acetates are likewise made by acting upon thin sheets of copper, in small vessels, with acetic acid. The copper is not immersed in the acid, but suspended over it, so as nearly to touch its surface; a temperature of about 150° to 180° Fahr. (66° to 83° C.) is maintained during the operation. The plates become in time coated with acetate, which is scraped off and dried for the market, as in the forementioned processes.

The composition of blue verdigris, according to Berzelius and Phillips, is:—

	Dibasic Acetate calculated.	Berzelius.	Phillips.		
			French Verdigris.	English.	
				Crystallized.	Compressed
Cupric Oxide, ...	43·24	43·34	43·50	43·25	44·25
Anhydrous Acetic Acid,	27·57	27·45	29·30	28·30	29·62
Water, ...	29 19	29·21	25·20	28·45	25·51
Impurities, ...	..	..	2·00	..	0·62
	100·00	100·00	100·00	100·00	100·00

This salt is employed in calico printing for precisely the same purpose as the neutral acetate, namely, as a resist paste in the indigo dye bath. All the acetates of copper are used for painting in oil. In medicine they are employed as escharotics. They are very poisonous; their manufacture, however, does not appear to be productive of inconvenience to the workpeople engaged.

Good verdigris should be dry, have a fine bluish-green colour, and be soluble in dilute acetic and sulphuric acids, and also in ammonia.

Verdigris is often adulterated, generally with finely ground pumice, chalk, and sulphate of copper. The purchasers acquainted with this article judge of the relative purity of the sample from its bright colour, and by kneading it on the palm of the hand with a little water; by the latter test the presence of sand is detected, as the acetates when alone form a paste free from any grittiness.

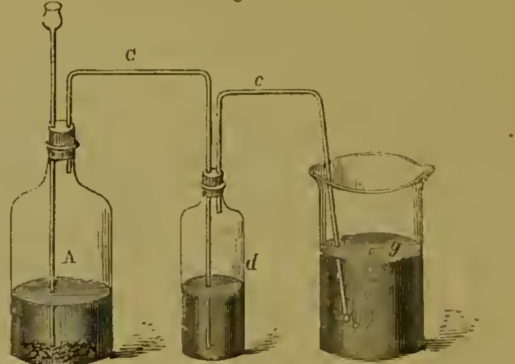
For the detection of chalk—carbonate of lime—a weighed portion of the verdigris, in powder, is introduced into a flask, and hydrochloric acid poured upon it; if effervescence takes place, it indicates the presence of carbonates; should, however, no effervescence occur, and at the same time a residue of silica remains undissolved, it shows that pumice, or some analogous body, is the adulterant. The residue is greater or less in proportion to the extent of adulteration.

If the hydrochloric acid solution of the above be filtered off, and the residue, being well washed, dried in a water-bath at 212° Fahr. (100° C.), and afterwards burned in a weighed platinum crucible till the whole of the charcoal of the paper is consumed, and weighed—the increase of weight will give the amount of insoluble impurities in the sample.

Sulphates of copper or iron are detected by adding barium chloride to the filtered liquid and washings; if either of these is present, a white insoluble precipitate of barium sulphate will fall down. By placing the beaker on a hot sand-bath the precipitate quickly settles down, after which it is filtered off, washed with boiling-hot water as long as any barium chloride is extracted, and then placed in a water or air-bath to dry: the precipitate is next detached from the filter upon a sheet of clean dry glazed paper; the filter is burned alone in the platinum crucible till the ashes contain no charcoal from the paper; the precipitate is then introduced, heated to redness, and when cold weighed.

The amount of the chalk or other lime compound may be ascertained by weighing out 100 grains, and dissolving them in a beaker-glass with hydrochloric acid; the clear solution is then filtered off from the residue, and a current of sulphuretted hydrogen passed through the liquid, till the whole of the copper is thrown down in the form of a brownish-black precipitate. The sulphuretted hydrogen—sulphydric acid—is generated in an apparatus like the annexed Fig. 34:—a is a bottle containing pieces of iron sulphide, and a few ounces of water; through the cork two tubes pass; the funnelled one, b, reaches to the bottom, and the other, c, opens at a quarter of an inch below the cork, which should fit the bottle air-tight. The sulphuretted hydrogen is generated by pouring through the funnel tube, b, some strong sulphuric acid, which reacts on the iron sulphide, giving rise to the gas, which passes off by the tube, c, bent at right angles, the longer limb passing through the cork of the bottle, *d*, which contains some distilled water, for the purpose of washing the gas. A second tube, *c*, bent at right angles, conveys the gas to the beaker-glass, *g*, containing the solution, from which the substance is to be precipitated.

Fig. 34.



The subjoined equation exhibits the decomposition:—

$$\overset{\text{Iron sulphide.}}{FeS} + \overset{\text{Sulphuric acid.}}{H_2SO_4} = \overset{\text{Iron sulphate.}}{FeSO_4} + \overset{\text{Hydrogen sulphide.}}{H_2S.}$$

When the whole of the copper has been precipitated as sulphide, it is filtered off, washed with water, and the washings added to the filtrate. Ammonia, in slight excess, is poured into the solution, and then ammonium oxalate, and the beaker-glass placed on the sand-bath. When the lime oxalate has subsided, it is collected, washed with water, dried as the preceding precipitates, and burned in a platinum crucible. The ignition should be gentle, and at a heat very little above dull redness, so as not to expel carbonic acid from the carbonate of lime, which is formed by the action of the heat on the lime oxalate, as is shown in the annexed decomposition:—

$$\overset{\text{Calcium oxalate.}}{CaC_2O_4} = \overset{\text{Calcium carbonate.}}{CaCO_3} + \overset{\text{Carbonic oxide.}}{CO}$$

The weight of the carbonate of lime obtained is equal to the amount of that adulterant which has been added to the verdigris.

Verdigris generally contains about 3 per cent. of impurities. Sometimes the insoluble matter in it is 6 per cent.; in such a case, however, the article is of inferior quality.

Formerly the manufacture of verdigris was one of the most lucrative in Belgium, and it was also carried on profitably in France; but at the present time the production of this substance is not confined to those countries. France still produces considerable quantities of the article, and nearly the whole of the salt imported into this kingdom is from that country. Until the 19th of March, 1845, the duty on verdigris imported into this country was 1$\frac{1}{20}$d. per lb. weight; it was then subjected to an *ad valorem* duty of 10 per cent., which was repealed in 1853. The imports are nevertheless small, as large quantities of verdigris are prepared in England with pyroligneous acid and cider refuse.

IRON ACETATES.—There are two acetates of iron, ferrous acetate and ferric acetate.

Ferrous Acetate, $Fe(C_2H_3O_2)_2$.—This salt may be prepared by dissolving either iron sulphide or metallic iron (generally iron turnings are used) in strong acetic acid; in the former case sulphuretted hydrogen, in the latter gaseous hydrogen, is evolved.

$$\overset{\text{Ferrous sulphide.}}{FeS} + \overset{\text{Acetic acid.}}{2C_2H_4O_2} = \overset{\text{Ferrous acetate.}}{Fe(C_2H_3O_2)_2} + \overset{\text{Sulphydric acid.}}{H_2S.}$$

$$Fe + \overset{\text{Acetic acid.}}{2C_2H_4O_2} = \overset{\text{Ferrous acetate.}}{Fe(C_2H_3O_2)_2} + 2H.$$

On concentrating the solution, small colourless silky prismatic needles are obtained, which are very soluble in water, and which, when exposed to the air, rapidly absorb oxygen, and become converted into ferric acetate. This compound may likewise be obtained by decomposing a solution of the ferrous sulphate (green vitriol, $FeSO_4$) by calcium or lead acetate: 1615 parts of ferrous sulphate require 2575 parts of lead acetate, or 999 parts of calcium acetate; but in this method a greater or less amount of ferric acetate is also formed, which can, however, be reduced to the state of ferrous acetate by passing a stream of sulphuretted hydrogen gas through the liquid.

The avidity with which this substance absorbs oxygen renders it of great value as a reducing agent.

For commercial purposes this compound is manufactured as follows:—Into a large cast-iron boiler or pot a quantity of iron turnings, hoops, or nails are introduced, and acetic acid—the crude pyroligneous acid from the distillation of wood—is poured in upon them. The strength of the acid is generally of 7° Twaddle, or spec. grav. 1·035. A temperature of 150° Fahr. (66° C.) is maintained till the solution of ferrous acetate is obtained, of a spec. grav. 1·09, or 18° T., at 60° Fahr. (15° C.). During the solution of the iron much tarry matter separates, which is skimmed off, and the solution frequently agitated, to free it as much as possible from the tar. As soon as the above strength is gained the solution is allowed to cool, for a further quantity of impurities to separate. When clean turnings are operated upon, the process of solution is completed in five to seven days. The hydrogen that is eliminated during the solution of the iron prevents the oxidation of the iron salt, as is seen in the equation already given. Were any ferric acetate formed, the hydrogen, by combining with part of its oxygen, would again reconvert it to ferrous acetate.

Some printers dissolve the iron without the aid of heat, but this method is slow and unsatisfactory, for the deposit of tarry bodies on the iron prevents the action of the acetic acid; besides, from the long exposure to the air, some sesquisalt of iron is generated. The usual produce from 100 gallons of acetic acid, and a proportionate quantity of iron turnings, is 60 to 70 gallons of acetate of iron, spec. grav. 1·090, or 18° T.; and when this solution is reduced by the addition of water to a spec. grav. 1·060, it produces with madder a deep black.

The mordant is likewise made by decomposing a solution of sulphate of iron ($FeSO_4$) by acetate of lime; the proportions employed are the following:—

400 lbs. of sulphate of iron—copperas—dissolved in
100 gallons of hot water, and the solution decomposed in
75 gallons of acetate of lime liquor, spec. grav. 1·08.

On agitating the mixture, the decomposition is rendered complete; the clear liquor, which is siphoned off after subsidence of the sulphate of lime, possesses a density of 1·110.

When the liquor is not required for immediate use it becomes oxidized, and deposits ferric acetate; to prevent this some metallic iron is left in contact with the solution. Sometimes, when a large quantity of pyroligneous matters deposits in the solution, the iron is prevented from acting by becoming coated with these substances; in such instances the formation of the basic acetate may be prevented by suspending fine iron wire in the liquid.

In some of the continental factories the ferrous acetate is manufactured by decomposing the carbonate of iron (ferrous carbonate, $FeCO_3$) with lead acetate: lead carbonate precipitates, and the blackish supernatant liquor is the acetate of iron in a very pure state. It is kept from oxidizing by immersing in it some bright iron filings. The lead-salt formed repays the cost of the manufacture of the acetate.

Both ferrous and ferric acetates are precipitated by sulphuretted hydrogen.

Ferric Acetates.—*Neutral Ferric Acetate; Sesquiacetate of Iron*, $Fe^{vi}_2(C_2H_3O_2)_6$.—This salt may be obtained in the pure state by decomposing a solution of lead acetate by addition of ferric sulphate ($Fe^{vi}_2(SO_4)_3$) in slight excess. In the course of twenty-four hours the excess of ferric sulphate precipitates as a basic salt. It is also produced, though more slowly, by dissolving ferric hydrate or ferric carbonate, obtained by precipitation, in strong acetic acid. This method occupies more time, but affords better guarantees for the purity of the compound. For ordinary purposes it may be made by mixing solutions of ferric sulphate and calcium or barium acetate in an iron vessel, and agitating the liquid.

The acetate thus obtained by the use of cold liquors is wine-red in colour, and presents all the characteristics of solutions of ferric salts. It is uncrystallizable, and on evaporation yields a deliquescent gelatinous paste. It is also soluble in alcohol.

When a solution of ferric acetate is brought nearly to the boiling point its colour becomes four or five times more intense, and it evolves a distinct odour of acetic acid, without, however, producing any precipitate. The salt has nevertheless become more basic, and addition of any soluble sulphate, or even of free sulphuric acid, immediately precipitates the whole of the iron as ferrous sulphate ($FeSO_4$). After cooling, this basic acetate is stable even in presence of excess of acid, for it retains its colour, and can be precipitated by sulphuric acid at the end of several hours. If the solution is allowed to rest for a few days neutral acetate, with its usual characteristics, is regenerated.

When a solution of ferric acetate is boiled rapidly it disengages acetic acid vapours, and in about half an hour's time begins to separate its iron in the state of hydrate (probably $Fe_2O_3,2H_2O$).

If in place of boiling the solution in an open vessel, it is heated in a closed vessel placed in a water bath to 212° Fahr. (100° C.) for a few hours, it undergoes a very remarkable change. Its colour becomes, little by little, of a brighter red colour, without losing in intensity; seen by reflected light it appears opaque and opalescent, but appears perfectly clear and limpid when viewed by reflected light, even when examined under the microscope. It has also lost the metallic taste peculiar to salts of iron, and rather resembles vinegar in its flavour. If the exposure in the water bath has been sufficiently prolonged, addition of potassium ferrocyanide produces no precipitate, nor does the sulphocyanide augment its red colour. A trace of sulphuric acid or any alkaline salt suffices to precipitate the whole of the iron in solution as a ferric hydrate of a red colour, and totally insoluble in all acids in the cold. Nitric and hydrochloric acids, when concentrated, precipitate ferric hydrate in the form of a granular brown powder, which dissolves completely in cold water, reproducing a solution, red in colour, and transparent to transmitted, but opaque to reflected light. This peculiar characteristic of the brick-red liquor just described is held by Péan de Saint Gilles, who first observed the phenomena, to be due, not to a chemical, but to a purely mechanical action exercised by the acetic acid upon the ferric hydrate; separating its insoluble particles to the minutest state of division, and thus simulating a true solution. Many chemists explain in this manner the apparent solution of Prussian blue in oxalic acid.

Solution of ferric acetate is much used as a mordant by dyers; like the acetate of alumina, it deposits an insoluble basic salt when heated, and hence its utility in dyeing operations.

Triferric diacetate—$Fe^{vi}_2\left\{\begin{matrix}(C_2H_3O_2)_2\\(H_2O)_4\end{matrix}\right\}2Fe_2O_3$—is an insoluble yellow powder, precipitating from ferrous acetate which has oxidized in the air, and even out of the neutral ferric acetate when kept for some time, especially if an alkaline salt be present.

Sesquiacetate of iron is manufactured chiefly for the use of dyers, the salt being rarely employed by the calico-printer.

Where the dyer requires uniform grounds, he cannot well employ ferrous salts, for when cotton is impregnated with such a solution as copperas or the ferrous acetate, it attracts oxygen from the air while drying; and the sesquioxide of iron, or a basic salt, collects in those parts not yet dry, and will, of course, produce darker spots in those places when the cloth goes in the dye-bath. It is therefore better to prepare a ferric acetate at once, either by pouring acetic acid repeatedly over iron turnings for several weeks, in vessels exposed freely to the air, or, what is still better, by double decomposition of a ferric salt with lead acetate or lime acetate.

A recipe for this purpose states:—Dissolve 1 lb. of iron-alum in half a gallon of water, add 1 lb. of acetate of lead, stir the liquor well, let it settle, and decant or filter. The solution made from iron-alum will not keep long, as it gradually deposits an insoluble basic salt from the presence of potassium sulphate, while that made from ferric sulphate will retain its properties for a great length of time; but on the other hand, iron-alum is more convenient to use, from its containing known quantities of ferric oxide. The difficulty above-mentioned may be obviated by preparing only so much solution as is required for immediate use.

Ferric acetate may also be made from ferric sulphate and lead acetate. As the ferric sulphate is not so uniform in composition as iron-alum, it may be well to ascertain how much oxide of iron and sulphuric acid it contains, in order to know what quantity of lead acetate to employ in its decomposition. For this purpose weigh out 100 grains; dissolve in water, and filter; add 285 grains crystallized acetate of lead dissolved in water, filter, and weigh the precipitate, which is sulphate of lead: every 76 grains of this sulphate of lead require 95 or more (safely, 90) grains of lead acetate to insure sufficient decomposition. Calling grains pounds, the operation may then be conducted on a large scale. The excess of lead acetate in the acetate of iron mordant may be ascertained by diluting a little of the clear liquor with water, and adding a few drops of sulphuric acid; if it becomes cloudy, there is an excess of lead acetate which may prove injurious to colours, but this is easily obviated by adding ferric sulphate until the clear liquor is no longer affected by sulphuric acid. For the ordinary operations of the dyer it may not be necessary to decompose all the sesquisulphate of iron; but for printing, and particularly for full russets, the salt with which the goods are charged should be wholly ferric acetate, since if sulphate was contained some of it would disappear in washing the goods, whilst the basic acetate of iron cannot be removed by water.

The acetates of iron are employed in woollen dyeing to produce blue with potassium ferrocyanide (K_4FeCy_6), yellow prussiate; in cotton dyeing and printing, and in silk dyeing, they are used for blacks, russets, &c. Ferrous acetate is used with madder, for violet; or together with red liquor, for brown; it is also used for dyeing hats and furs black, and for blackening leather, wood, &c. Some dyers prefer the ferrous acetate, because, by the oxidation of the iron subsequent to dyeing, the colours are more resistant; but greater uniformity of the ground is insured by the use of ferric acetate.

An iron mordant containing a mixture of ferric and ferrous acetates is largely used; it is made by pouring pyroligneous acid on iron turnings in a series of vessels placed obliquely one above the other—as will be more particularly described under acetate of lead—suffering the acid to remain some length of time in contact with the metal, and repeating the operation until the acid is saturated with iron. This is the method now almost universally employed for the manufacture of these valuable mordants.

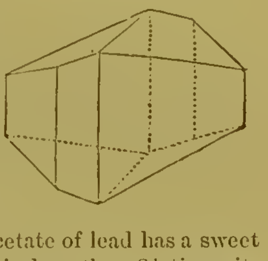
LEAD ACETATES.—These are the following:—

Neutral acetate or diacetate,...	$Pb(C_2H_3O_2)_2$.
Sesquibasic acetate,..........	$2(Pb(C_2H_3O_2)_2),Pb(HO)_2$.
Dibasic acetate, ...	$2(Pb(C_2H_3O_2)(HO)) + H_2O$.
Tribasic acetate,.............	$Pb(C_2H_3O_2),2PbO,H_2O$.
Sexbasic acetate,.............	$Pb(C_2H_3O_2)(HO),2PbO$.

Neutral Acetate, Diacetate, Sugar of Lead, Salt of Saturn; Sucre de Saturne, French; *Bleizucker*, German; $Pb(C_2H_3O_2)_2$.—As may be seen, lead unites with acetic acid in various proportions; the most important of these combinations is the normal salt, known in commerce as sugar of lead. It is used to a great extent in the calico-printing business, and likewise in dyeing, for the preparation of other compounds employed in those trades, and occasionally as a medicinal agent, and in pharmaceutical preparations.

It may be prepared by digesting litharge, monoxide of lead (PbO), in dilute acetic acid, or by exposing thin sheets of lead in a chamber to the joint action of air and the vapour of acetic acid. The lead becomes corroded, and a mixture of carbonate and acetate of lead is formed on the surface of the sheets. When this coating becomes thick enough it is scraped off, and dissolved in a slight excess of acetic acid. On evaporating this solution until it attains the density of 2·2, the lead acetate crystallizes, if the hot solution be set aside to cool rapidly, in clusters of fine needles; but if the evaporation be conducted slowly, the crystals are truncated and flattened quadrangular and hexahedral prisms (Fig. 35), derived from a right rhombic prism. The crystals are permanent in the air, but are apt to effloresce and become anhydrous if the temperature ranges between 70° and 100° Fahr. (21° and 38° C.). Anhydrous acetate of lead is soluble in boiling absolute alcohol, and is deposited again in hexagonal plates on the slow cooling of the spirituous solution. Acetate of lead has a sweet astringent taste, is soluble in less than 3½ times its weight of boiling water, and in nearly the same quantity of cold water. PAYEN states that 100 parts of water at 60° Fahr. dissolve 59 parts of the crystallized acetate of lead. Its solution reddens litmus. The crystallized salt is fusible in its water of crystallization at 167° Fahr. (75° C.); just below 212° Fahr. (100° C.) it loses its water and part of its acid, and becomes converted into a white laminated mass of sesquibasic acetate.

Fig. 35.



On raising the temperature higher the substance fuses, is completely decomposed, and evolves all the compounds usually obtained in the destructive distillation of the acetates of the heavy metals, leaving a residue of metallic lead, in a very minute state of division, with some charcoal. When this distillation is conducted in a glass tube closed at one end, and having the other drawn out for convenience of sealing at the end of the operation, the well known lead pyrophorus is made. The particles of metallic lead are so small that when thrown into the air oxygen molecules come into such intimate contact with them, that ignition is effected from the rapidity with which lead oxide is formed.

A slight decomposition occurs when the neutral salt is exposed to an atmosphere of carbonic acid—carbonate of lead being formed; the portion of acetic acid thus liberated protects the remainder from further change.

KRAFT prepares lead acetate by treating 100 parts of lead sulphate with 52 parts of calcium acetate or 84 parts of barium acetate. In the cold the reaction is complete in a very short time; when the mixture is heated the change is instantaneous.

Brown Acetate of Lead.—Distilled pyroligneous acid is saturated with litharge in a tub, and the muddy solution ladled out into a large tun to settle; the solid matter quickly subsides, and the clear solution is then transferred into either a cast or wrought iron pan, about 6 feet long by 4 feet broad. The solution is here brought to the boiling point and then allowed to settle; it is next transferred into a large hemispherical pan capable of holding 300 or 400 gallons, where it is evaporated down to about crystallizing strength. When the solution has become dense enough to crystallize, about three times its bulk of water is run in upon it whilst boiling, the solution being constantly stirred. By this treatment the impurities are disengaged, and may be skimmed off as fast as they rise to the surface; after they are removed the evaporation goes on as before. If the solution be still too much coloured, another dose of water must be given. A little practice soon enables the operator to know when the evaporation should be checked. The ordinary method used by the workman is to rinse the skimming ladle through the liquid, and holding it above the pan, observing how many drops fall from it before the solution takes a stringy appearance; if only ten or twelve fall, then it is sufficiently strong. The liquid is now ladled out into wrought-iron pans to crystallize; the pans are 5 feet long by 3 feet broad, and about 6 inches deep, the sides being bevelled or sloping outwards from the bottom. After the crystals have become sufficiently firm, the sugar of lead is taken out by inverting the pan on a cloth. The pots used in the above process are heated only at the bottom.

White Acetate of Lead.—This is prepared by dissolving litharge in acetic acid. The acetic acid is introduced into a vessel, the litharge added by degrees, and the menstruum kept in brisk commotion after each addition, until the solution only slightly reddens litmus paper; a quantity of water, equal to about one-half of the acid employed, is then run into the lead solution; heat is applied, and the mixture slowly evaporated for about twelve hours, or until it has acquired a density of about 1·500. During evaporation any impurities which rise to the surface are skimmed off, and when the solution has acquired its proper gravity it is drawn into the crystallizing pans. When the crystals have become sufficiently hard to allow of their being taken *en masse* from the crystallizers, they are drained and placed on wooden racks in the drying-house, and, when dry, cleaned and broken up into fragments for the market.

The mother liquor, which contains neutral and basic acetates of lead and other metallic salts, may either be treated with vinegar, evaporated, recrystallized, and the residue employed as washings in subsequent operations; or it may be decomposed by the carbonates of soda or lime, and used as carbonate of lead; or dissolved in acetic acid, and the supernatant acetate of soda or lime recovered.

The vessels used in this manufacture are, in most cases, of lead. In Wales the mixing pans are of lead, three-quarters of an inch thick, 7 feet long by 4½ feet wide, and 1 foot deep. These pans are set on iron plates over arches, and the fireplaces are outside the building, in order that the acetate may not be discoloured by the smoke, &c., from the coal. The crystallizing pans are of wood, lined with thin copper, and are about 4 feet long by 2 feet wide, and from 6 to 8 inches deep, sloping inwards at the edges. At Pitchcombe both the mixing and crystallizing vessels are of copper, having a strip of lead soldered down the sides and across the bottom of the vessel, with the idea of rendering the metal more electro-negative, so as to prevent the acetic acid from acting on it. Very great care is requisite in the drying of the sugar of lead; the temperature of the desiccating house should not exceed 90° Fahr. (32° C.). In Wales the heated air of a stove, placed outside the drying-house, is conveyed through pipes passing round the interior; at other places steam heat is the agent for this purpose, which is much to be preferred on account of its being more easily regulated.

That the manufacturer may the better judge of the success of his operations from the amount of the product obtained, the following will serve as a precedent:—112 lbs. of good Newcastle litharge should produce 187 lbs. of sugar of lead, when treated with 127 lbs. of acetic acid of spec. grav. 1·057, but not more than 180 lbs. are obtained in practice. In a factory in Wales a ton of Welsh litharge produces, with the acid obtained from one ton of acetate of lime, from 28 to 30 cwts. of sugar of lead; and in another manufactory, one ton of best Newcastle litharge, with the acid from 1½ ton of the acetate of lime, produces 33 cwts. of the lead salt.

The following process with metallic lead, recommended first by **Berard**, is easily executed, and is said by **Runge** to yield a good product with great economy. Granulated lead, the tailings in the white lead manufacture, &c., are put in several vessels—say eight—one above the other, upon steps, so that the liquid may be run from one to the other. The upper one is filled with acetic acid, and after half an hour let off into the second, after another half hour into the third, and so on to the last or eighth vessel. The acid causes the lead to absorb oxygen so rapidly from the air as to become hot. When the acid runs off from the lowest, it is thrown on the uppermost vessel a second time, and carries off the acetate of lead formed: after passing through the whole series the solution is so strong that it may be evaporated at once to crystallize. There are two points of importance in this manufacture. Whatever method may be pursued, a strong acid should be employed, in order that less of it may be lost in concentrating the liquid, that time may be economized, and an acid reaction retained in the liquid, to prevent the formation of a basic salt.

It may not be amiss to call attention here to a process, patented many years ago, for preparing acetate of lead and other acetates, and for which fresh patents have recently been taken out. It consists in employing the acid in the state of vapour, to act upon the bases, instead of using it in the liquid form. A vessel is provided of adequate capacity for the quantity of acetate required, and constructed of such material as will not be readily destroyed by the acid. The top of this vessel is closed hermetically by a cover, fastened down by any convenient means, and in the lower part of the vessel is placed either a minutely-perforated false bottom, or a coiled tube of several convolutions, minutely perforated, to permit vapour to permeate freely. To prevent the loss of acid there is also placed, at different degrees of elevation, several perforated diaphragms, similar to the false bottom just mentioned, on each of which is spread a layer of litharge, after which the cover of the vessel is to be accurately closed. By means of an ordinary distillatory apparatus liquid acetic acid—strong or weak, pure or impure—is converted into vapour, which is then conducted by means of a pipe into the convoluted and perforated one before mentioned, or between the real bottom of the vessel and the perforated false bottom; hence the acetic acid, passing through the numerous perforations of the false bottom and diaphragms, diffuses itself throughout every part of the vessel, and forms the acetate of the particular metal used, which falls to the bottom of the vessel, in its descent meeting with the ascending streams of vapour, the acid of which renders it perfectly neutral. Meanwhile the more aqueous part of the vapour ascends, and in its passage through the successive layers of the base is deprived of its remaining acid, and at last as simple steam is allowed to escape through pipes at the top of the vessel. As this steam still maintains a boiling temperature, it is conducted through a worm and used to evaporate the acetate or mother liquor. Distillation of the acid is continued until the acetate in the vessel has acquired the proper degree of concentration for crystallization, which is easily ascertained by examining a small quantity drawn off by a tap at the bottom of the vessel, through which the whole contents are discharged when the operation is completed. As the process draws to its close the vapour issues from the vessel charged with a certain portion of acid; and that no loss may be sustained by its escape into the atmosphere, it is conducted into another vessel prepared like the first-mentioned, so that every particle of the acid issuing from the first vessel may be absorbed. The second vessel is then made the first, and the other recharged and made the second. As the temperature of the solution of the acetate can never exceed that of the vapour, the crystalline product is of fine quality.

Lead Sesquibasic Acetate, Triplumbic Tetracetate, $2(Pb(C_2H_3O_2)_2),Pb(HO)_2$.—This salt is obtained by heating the diacetate until it becomes a white porous mass; this is redissolved in water and set aside to crystallize. Sesquibasic acetate is soluble in both water and alcohol; its solutions are alkaline.

Lead Dibasic Acetate, Plumbic Monoacetate, $2(Pb(C_2H_3O_2)(HO)) + H_2O$.—One equivalent of lead diacetate is boiled with one equivalent of lead oxide; on cooling the salt crystallizes—

$$Pb(C_2H_3O_2)_2 + PbO + H_2O = 2(Pb(C_2H_3O_2)(HO)).$$

Lead Tribasic Acetate, Triplumbic Diacetate, $Pb(C_2H_3O_2)_2,2PbO,H_2O$.—Prepared by digesting at a moderate heat 7 parts of finely powdered litharge, 6 parts of lead acetate, and 30 parts of water: or into 100 volumes of boiling water is poured 100 volumes of aqueous diacetate solution, saturated at 86° Fahr. (30° C.), and afterwards a mixture of pure water at 140° Fahr. (60° C.), with 20 volumes of ammonia liquor free from carbonate. The vessel is then immediately closed, and in a short time abundance of tribasic acetate crystallizes out. This salt presents itself under the form of long needles. It is insoluble in alcohol, very soluble in water, its solution being alkaline. Tribasic acetate is the most stable of all the subacetates of lead. It takes a leading part in the manufacture of white lead by Clichy's process; it is, in point of fact, a solution of this salt which is decomposed by the carbonic acid and gives rise to the carbonate of lead, being itself at the same time converted into lead diacetate. In the Dutch process the formation of lead carbonate is, according to Pelouze, also due to the formation of triplumbic diacetate on the surface of the sheets of lead, which is, in its turn, decomposed by the carbonic acid.

Lead Sexbasic Acetate, Triplumbic Monoacetate, $Pb(C_2H_3O_2)HO,2PbO$.—This body is prepared by digesting any of the preceding salts with lead oxide. It is a white powder slightly soluble in boiling water, from which it crystallizes out in silky needles, which consist of two equivalents of the salt combined with three equivalents of water.

Acetum Saturni consists principally of triplumbic monoacetate; it is prepared by boiling 9 parts of water with 3 parts of lead diacetate and 1 part of lead oxide. *Goulard water* or *lead vinegar* is an aqueous solution of this preparation: on account of its power of coagulating mucus, &c., it is much used for forming a skin over wounds and sores.

Solution of lead subacetates are used in chemistry for the precipitation of colouring matters, tannin, gums, &c. Guignet has observed that a solution of basic acetates of lead produces distinct precipitate in a liquor containing only one per cent. of a soluble nitrate.

All solutions of basic acetates of lead absorb carbonic acid with great avidity, and become turbid from the precipitation of lead carbonate; their reactions with litmus and turmeric are alkaline.

Calcium Diacetate; *Acetate of Lime.*—This salt is formed in the manufacture of acetic acid when lime carbonate is dissolved in acetic acid liquors. The reaction is—

$$CaCO_3 + 2C_2H_4O_2 = Ca(C_2H_3O_2)_2 + H_2O + CO_2.$$

According to Pelouze, carbonate of lime does not dissolve in the monohydrated acid. The pure salt crystallizes from alcoholic solutions in silky, acicular prisms, containing one equivalent of water ($Ca(C_2H_3O_2)_2 + H_2O$), they have a bitterish saline taste, and effloresce when heated to 212° Fahr. (100° C.); they are soluble in water and alcohol. The dry salt has the property of being phosphorescent in the dark, when triturated at 226° Fahr. (108° C.).

A curtailed account of the production of this compound was given when describing the preparation of pure acetic acid from pyroligneous acid, and likewise of the acetate of soda. A modification of the process, in order to produce a purer article for the market, will be now considered. The following is the mode of working in large factories:—The crude acid liquor from the distillation of wood, after separating the pyroxylic spirit, is either distilled or run off into other convenient vessels, according as the *grey* or *brown* acetate is to be the resultant product. In either case the subsequent procedure is the same: 500 or 1000 gallons of the liquid are run off into wooden or iron vessels, of suitable capacity, and powdered chalk or slacked lime added, till a slight excess remains undissolved, and the whole agitated briskly for some time, in order to insure complete combination. The mixture is then allowed to rest at a temperature of 150° Fahr. (65° C.), till the excess of lime and tarry compounds subsides, when the clear supernatant liquid is siphoned off into the evaporating pans. These are, in most factories, wooden vessels lined with lead, and heated by coils of iron pipes placed within them, through which steam passes; occasionally they are shallow, and made of sheet-iron, and placed together directly over the fire. The solution of the lime salt is kept simmering, and briskly agitated during the evaporation, and the scum of tarry impurities that agglomerates at the surface constantly skimmed off. As soon as the acetate of lime begins to form it is separated from the liquor by the skimmers, and thrown into wicker baskets suspended over the pans, so that the solution draining from the salt may not cool.

The subjoined results were obtained by the use of three sheet-iron pans of about 18 inches in depth, each capable of containing 450 gallons of the solution:—

	Number of gallons of liquor evaporated.		Producing of dry acetate of lime.
In the first six days, of twenty-four hours each,..............	7,020	....	73 cwt.
In the second six days, of twenty-four hours each,..............	8,060	....	92 "
In the third week of six days, of twenty-four hours each,.......	7,000	....	78 "

Two of the pans contained *brown* acetate of lime liquor.

The yield of acetate here mentioned is of course dependent upon the variety of wood submitted to distillation, as also upon its state of dryness and the proper regulation of temperature. That part of the process which demands the greatest attention is the *drying*, as on its proper execution the success of the operation in a great measure depends. Many methods are in use for drying the lime salt, some of which are very unscientific. In some factories the salt is dried by spreading it in thick layers on the top of the brickwork surrounding the carbonizing retorts and steam-boilers; of course, in large works, where 10,000 to 15,000 gallons of liquor are evaporated weekly, the products cannot be dried in this way. When working on so large a scale it is customary to have a drying-house set apart for the purpose, and when the lime is burned on the premises the heat from the kilns is commonly used to dry the lime acetate, by conducting the products of combustion through flues in the floor of the drying-house. As a rule, there should be a drying-house attached to every factory, since the want of it may entail the loss of the whole of the acetic acid produced by the distillation of the wood.

The ordinary drying apparatus is a simple wind furnace, 7 or 8 feet long and 4½ feet broad, built of brick. At 6 inches above the ground is the ashpit, 8 inches broad and 12 inches high, which is covered with a grate of bricks. The fireplace is 20 inches high and 10 inches broad at the grate; over it is an arch of bricks, so that the fire cannot play on and heat very highly the iron drying-plate lying on the side of the hearth. The space below the drying-plate is separated from the hearth by a partition of bricks 3 or 4 inches high; 12 inches above the outlet of the hearth is a layer of iron bars 1½ to 2 inches from each other, and upon these is deposited the drying-plate. This consists of cast iron a quarter of an inch thick, and is formed according to the size of the furnace. Round the plate the furnace is built up to the height of 10 inches on the side of the front wall, leaving room for doors, which may be calculated at 2½ feet. These doors are two, one above the other, through which the whole interior of the furnace can be inspected. They are formed of plate iron, and have in their middle a sliding door to admit of the exit of the vapour of the acetate of lime and of some ventilation. A wall built at the end of the plate, or a clay partition, separates the whole of the drying-plate from the chimney. In the walls of the furnace iron bars are fixed, and upon these a second drying-plate covers the drying space. This plate, as it does not come in contact with the fire, may consist of good iron or of clay. Above this drying space another is formed by means of the chimney. The hot air passes both under and above the drying space, and thence into the chimney, which is situated at the side of the furnace, and can be shut by a valve. The prevailing temperature in the drying room is 167° to 235° Fahr. (75° to 112° C.).

When the whole of the furnace is heated to the proper temperature the fire is slackened. If wood be employed for fuel the sliding door should be opened at the commencement, in order to allow the moisture to escape. The salt is transferred from the baskets over the evaporating pans to the drying-plate, and spread out to the depth of 2 inches; and after the first portion has become somewhat dry the depth is increased to 4 or 5 inches; the heat, as already mentioned, is kept up for twenty-four hours, during which the salt is repeatedly turned. Subsequently, when the mass appears to be becoming dry, the temperature is raised to 257° Fahr. (125° C.), in order to expel every trace of water; care, however, must be taken that the heat is gradually applied, and that no vapours arise from the acetate of lime, for decomposition of the salt would then be taking place; neither should any spark of fire be suffered to come in contact with the dried salt, since it possesses the characteristic property of igniting and burning like sugar of lead. During the drying process the tarry and oleaginous matters with which the acetate is impregnated are decomposed, and a black charcoal remains, which appears in streaks through the dry mass. On dissolving the desiccated acetate of lime in 3 parts of hot water, and filtering through coarse animal charcoal or gravel, the charcoal and decomposed carbonaceous matters of the salt are separated; and the solution, upon evaporation to dryness, yields a very pure and nearly colourless product: 140 lbs. of this salt is the average produce of 1 ton of wood.

MANGANESE ACETATE, $Mn(C_2H_3O_2)_2$.—This compound is prepared by dissolving pure manganese carbonate ($MnCO_3$) in acetic acid, evaporating the solution, and crystallizing. The crystals are of the rhombic prism, and occasionally in plates of an amethystine colour; they are permanent in air, soluble in alcohol and in about three and a half times their weight of cold water.

On the large scale this salt is manufactured by precipitating a solution of the manganous sulphate by one of lime acetate, and agitating the liquor to decompose the whole of the manganese salt.

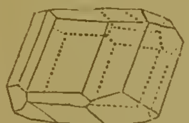
$$MnSO_4 + Ca(C_2H_3O_2)_2 = Mn(C_2H_3O_2)_2 + CaSO_4.$$

It sometimes happens that a portion of the manganese salt is not acted upon by the acetate of lime; in this case a concentrated solution of acetate of lead is employed towards the end of the process to effect the complete decomposition. The mixed precipitate of sulphate of lime and lead is filtered off, and the filtrate evaporated and crystallized, or, if deemed necessary, used directly. The best acetate of manganese is made by adding to 4 parts of manganous sulphate, dissolved in 3 parts of water, 7 parts of crystallized acetate of lead dissolved in 3 parts of water, agitating the solution, and drawing off the clear liquor for use.

Acetate of manganese is used in dyeing and calico printing, to give a brown colour to fabrics. Its principle of action depends upon the further oxidation of the manganese. The cloth is well steeped in a concentrated solution of the acetate of manganese, and printed; it is then passed through a bath containing bleaching powder, which oxidizes the manganese, producing a brown colour on that which, previous to its immersion in the bleaching or chloride of lime solution, was colourless.

SODIUM ACETATE, *Acetate of Soda; terre foliée minérale*, French; $NaC_2H_3O_2$.—This salt is formed by dissolving carbonate of soda in acetic acid, evaporating the solution, and setting the liquor aside to crystallize. The crystals are oblique rhombic prisms (Fig. 36) soluble in 3 parts of cold, in a less quantity of boiling water, and in 5 of alcohol. Their composition is $NaC_2H_3O_2 + 3H_2O$.

Fig. 36.



This salt possesses a pungent but not disagreeable taste. It is soluble in 3·9 parts of water at 44° Fahr. (6° C.), in 2·4 at 99° Fahr. (37° C.), and in 1·7 at 117° Fahr. (48° C.). According to GERARDIN, 100 parts of alcohol at 0·9904 dissolved 38 parts of the crystallized salt at 65° Fahr. (18° C.). The solubility diminishes if the alcohol is more concentrated; alcohol 0·832 dissolves only 2·1 parts. When this salt is exposed to dry air it loses its three equivalents of water, but regains them in a moist atmosphere. After being melted this salt is deliquescent, and takes up 7 equivalents of water; it then becomes a liquid supersaturated solution, which crystallizes with evolution of heat immediately a fragment of dry or crystallized or sodium acetate is thrown into it.

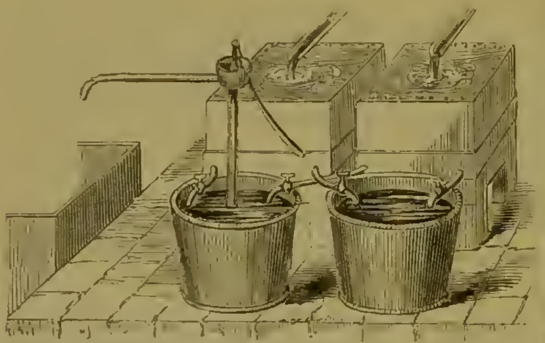
On the large scale the manufacture may be carried out in the following way:—A filtered solution of the common acetate of lime is precipitated by one of sulphate of soda at 98° or 100° Fahr. (36° to 38° C.), till the whole of the lime is thrown down as sulphate. (This process is not economical, on account of the formation of the double sulphate of calcium and sodium, $CaNa_2(SO_4)_4$.) The strength of the solution of the lime-salt is 1·116, and of the soda solution 1·250; or the sulphate of soda may be added in powder, and the whole well agitated until no further precipitation of sulphate of lime takes place, using the precaution, however, of adding the sulphate of soda sparingly, so as to prevent its excess. The mixture, after thorough agitation, is drawn off into a deep vessel and allowed to repose; and when the precipitated sulphate of lime has completely settled, the clear liquor is siphoned off and conducted to the evaporating pans for crystallization, the remaining lime-salt being washed with successive portions of water, till the whole of the acetate of soda is separated. The first washings may be added to the strong solutions in the evaporating pans, and the others retained for dissolving fresh portions of acetate of lime for subsequent decomposition. The process now most generally adopted is to distil the acetate of lime (after purification) with sulphuric acid, and saturating the acetic acid evolved with sodium carbonate.

Occasionally the solution of rough acetate of soda is filtered, and the precipitate washed in the following manner:—One or two backs are provided, according to the size of the factory; these are placed over two cisterns, each cistern being connected with both the backs by pipes branching from the latter to both cisterns; by means of a stop-cock in those connecting pipes the communication in either back may be cut off at will when required, as seen in Fig. 37. The backs are either square or circular, having false bottoms, in which are filters of stout twilled flannel. The charge from the decomposing pan is run on to one of the backs and filtered, till the whole of the strong liquor has passed into one of the cisterns; the connection with this cistern is then cut off, and when the washing of the residue commences the pipe communicating with the other cistern is opened, and conducts the washings into it. A fresh charge may then be introduced into the second back, and the pipe reaching to the cistern holding the strong liquor opened. Thus the strong liquor is always obtained by itself ready for evaporation. Another advantage is, that while the residue in one back is in the course of being washed, the other may be filtering off strong liquor. The washing is continued till the percolations are nearly tasteless; the residuum is dug out of the filters with wooden spades; iron spades cannot be used, as they are apt to cut the filters. The acetate of soda liquor is then pumped from the cisterns into the evaporating pans, which should be so constructed as to offer as much heated surface as possible, so that one man can keep their contents in brisk agitation. As the evaporation draws to a termination, much care and skill are required to keep the liquefied salt well stirred, so that every part may be equally under the influence of the heat, and decomposition of the whole mass prevented. The temperature should never rise higher than 500° Fahr. (260° C.), and in drying the acetate of soda 450° to 470° Fahr. (237° to 243° C.) is the temperature that should be applied. White fumes passing off from the fused mass are indicative of the decomposition of the salt; and if the fire be not checked immediately nothing will be left but carbonate of soda and charcoal.

If purified acetate of lime or pyroligneous acid be employed, the acetate of soda obtained by dissolving the above product in water, evaporating and crystallizing, will be quite pure.

Crystallization is conducted by dissolving the fused salt in twice its weight of water, and filtering the solution through filter-bags. The liquor is then evaporated till it acquires a spec. grav. of 1·50, when it is drawn off to the crystallizing pans. These may be made of boards lined with four-pound lead, 4 feet long, 2 feet wide, and 9 inches deep. The concentrated solution of the salt is left in the pans for two, three, or four days, according to the temperature of the room, till a crop of crystals are produced; the crystals are separated and deposited in baskets, where they are allowed to drain; the mother liquor is either returned to the evaporating pan, or is employed to dissolve fresh quantities of the crude salt. The crystals are washed with cold water, to separate any adhering mother liquor, and placed on shelves to dry, after which they are ready to be packed. The washings may be added to the mother liquor, and used as above.

Fig. 37.



The purification of this salt may also be effected in the following way:—The acetate of soda is dissolved in a large cylindrical lead vessel, heated by shooting steam into it. When the solution is completed it is run through a flannel filter into the top of a course of steamers, each furnished with a coil of three-quarter inch lead pipe. These vessels are made of four-pound sheet lead, cased in boards; the best size is about 24 feet long, 4 feet wide, and 9 inches deep. The pipe should be coiled from one end of the pan to the other, and should go up and down two or three times. Too much pipe cannot be used, as the rapidity of the evaporation depends upon the quantity employed. As the evaporation proceeds the liquor ought to be siphoned from the top to the second, and afterwards to the third steamer, and thus room made for more of the bulky weak liquor from the dissolving vessel. When a pellicle appears on the surface of the acetate solution it should be siphoned off into the crystallizers, and allowed to rest for two or three days, when beautiful crystals in oblique rhombic prisms will make their appearance. If large crystals are required the coolers are immersed in sawdust, or some other non - conducting material, and a longer time is allowed for them to form. In general cases the amount of acetate of soda obtained from 1 ton of the lime-salt ranges from 20 to 22 cwts. The impurities present in acetate of soda are sulphate of soda and acetate of lime; the presence of the latter is caused by the imperfect decomposition of the acetate of lime by sulphate of soda, and of the former by a too abundant use of that salt in decomposing the acetate of lime. The sulphate of soda is separated by skimming off the crystals of this salt, which form before those of the acetate of soda; but the acetate of lime cannot afterwards be got rid of, and is injurious in *running the crystals*, as the workmen term it; that is to say, it prevents crystallization. In some factories, instead of evaporating the acetate of soda to dryness, and subsequently roasting the dry mass, the purification is effected by repeated crystallizations and filtrations of the solution through animal charcoal, which has been previously washed with hydrochloric acid.

Sodium acetate has recently been used by Sacc with great success for the preservation of animal and vegetable substances. His method consists in the use of powdered acetate of sodium instead of common salt. To keep meat fresh, it is placed in a cask with layers of acetate of sodium interposed between the layers of meat in the proportion of one-fourth of the weight of the meat. In summer the action of the salt is immediate; in winter it is necessary to place the casks or barrels in a room heated to 58° Fahr. (44°·5 C.). As the salt abstracts the water from the meat, the cask is turned about. The operation is complete in about forty-eight hours, and the meat may then be packed with its pickle, or it may be dried in the air. If the casks are not full they may be filled up with a fresh pickle made by dissolving 1 part of acetate of soda in 3 parts of water. When the pickle is drawn off from the meat half the salt is deposited in crystals, and may be used again.

Meat which has been thus treated is prepared for cooking by steeping for at least twelve, and not more than twenty-four hours, according to the size of the piece, in tepid water, to which a small quantity of sal ammoniac has previously been added. This salt decomposes the acetate of sodium which remains in the meat, forming sodium chloride, or common salt, and ammonium acetate. The meat swells and resumes the colour and reactions of fresh meat. Animals, particularly fish and poultry, may be preserved entire for market purposes in a pickle of acetate of soda; the only precaution necessary being the removal of the intestines. Under the influence of the pickle the meat loses about one-fourth of its weight, and another quarter disappears when it is dried. The process is also said to be very well adapted to the preservation of vegetables. These generally lose thereby five-sixths of their weight. When needed for use, it is only necessary to soak them for twelve hours in water, and then cook them as if entirely fresh. It is necessary to scald vegetables until they lose their stiffness before applying the acetate of soda. After being twenty-four hours in the pickle at the temperature of about 86° Fahr. (30° C.), they are drained or pressed, and are then dried in the air. Potatoes must first be steamed, as they are not readily penetrated by the acetate of soda liquor. All articles of food prepared by this process should be kept in a dry place, as they would otherwise absorb moisture very quickly.

Potassium Acetates.—These are two in number—namely, the neutral acetate or monoacetate, and the acid acetate or diacetate of potassium.

Potassium Neutral Acetate, *Monoacetate of Potassium; Terra Foliata Tartari*, *Arcanum Tartari*, *Tartarus Regeneratus*, Alchemists; *blättererde*, *geblätterte weinsteinerde*, German; $KC_2H_3O_2$. — Acetic acid is present in the sap of very many plants, and is generally combined with potassium, forming neutral potassium acetate. When wood is calcined the potassium acetate is decomposed, the acetic acid being replaced by carbonic acid. It is by this interchange that the carbonate of potassium found in wood ashes is formed.

This salt may be prepared by any of the processes used to make sodium acetate. If prepared by dissolving carbonate of potassium in brown vinegar, Fremy advises that the carbonate be added in small portions at a time, and that care be taken to keep the liquor acid, by which means the formation of colouring matters by the action of the alkaline carbonate will be avoided.

Potassium monoacetate is generally deposited as a white foliated crystalline mass. The solution is evaporated to dryness, and cautiously fused, when the potassium monoacetate forms a white foliated crystalline mass. It is very soluble in water, and very deliquescent. According to Osann:—

	Fahr.	C.
1 part of potassium acetate dissolves in 0·531 parts of water at	36°	2°
1 part of potassium acetate dissolves in 0·437 parts of water at	57°	13·9
1 part of potassium acetate dissolves in 0·321 parts of water at	83°	28·5
1 part of potassium acetate dissolves in 0·203 parts of water at	144°	62°

Berzelius states that 0·125 parts of boiling water dissolves 1 part of salt.

Acetate of potassium is less soluble in alcohol than it is in water. From a concentrated boiling alcoholic solution it crystallizes out on cooling. It is precipitated from alcoholic solution in the cold by ether. Carbonic acid passed into the alcoholic solution decomposes the salt, forming carbonate of potassium and acetate of ethyl (acetic ether). When dissolved in acetic acid, it forms the acid salt, potassium diacetate.

Potassium acetate melts at 556° Fahr. (292° C.); at a red heat it is decomposed into acetone, hydrocarbons, empyreumatic products, and a residue of carbon and potassium carbonate.

Acetate of potassium, when heated with potassium hydrate in excess, becomes converted into carbonate of potassium and marsh gas:—

$$\overset{\text{Potassium acetate.}}{KC_2H_3O_2} + \overset{\text{Potassium hydrate.}}{KHO} = \overset{\text{Potassium carbonate.}}{KCO_3} + \overset{\text{Marsh gas.}}{CH_4}.$$

When heated with arsenious acid cacodyl is produced. This reaction is so decided, and the alliaceous odour evolved is so strongly marked, that it forms one of the best tests for small quantities of acetic acid.

Kolbe first obtained free methyl by the decomposition of acetate of potassium by electrolysis.

Potassium acetate is employed in medicine as a diuretic.

Chlorine gas dissolves in an aqueous solution of acetate of potassium, forming a powerful though unstable bleaching liquor; carbonic acid is evolved during the reaction.

Potassium Acid Acetate, *Potassium Diacetate*, $KC_2H_3O_2,C_2H_4O_2$.—This salt was first formed by Thomson, by evaporating an equivalent solution of potassium acetate in acetic acid in vacuo. Subsequently it was found by Melsius to be readily formed by dissolving acetate of potassium in an excess of acetic acid, and evaporating to dryness; the salt crystallizes in fine needles or oblate rhombic prisms, as the evaporation is rapid or the reverse.

Stannous Acetate, *Acetate of the Protoxide of Tin*, $Sn(C_2H_3O_2)_2$.—This salt is employed in calico-printing, to give light spirit colours to cloth. The process of manufacture is nearly the same as that for making sodium acetate. The proportions taken are, 103 parts of crystallized tin dichloride (stannous chloride, $SnCl_2$), dissolved in water, and 190 parts of crystallized lead acetate. It may likewise be prepared by dissolving tin oxide, or metallic tin, in acetic acid. In technical operations, the first method is usually followed. On evaporating the filtered solution to a syrupy consistence, and adding alcohol, the salt crystallizes in colourless transparent needles, which have a great tendency to oxidize, if at all exposed to the air.

Another recipe is to dissolve 30 lbs. of acetate of lead in 40 gallons of boiling water, and add 18¾ lbs. of crystals of chloride of tin; the mixture is then well stirred, allowed to settle, and the liquor filtered or siphoned off for use into casks or vessels, protected as much as possible from contact with the air. Some makers prefer to prepare the salt when immediately wanted for use.

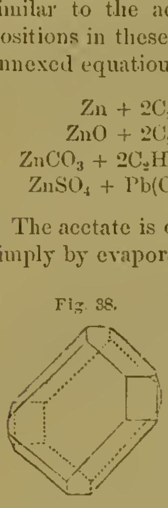
Hydrated stannic oxide (stannic acid, H_2SnO_3) also dissolves in acetic acid. On evaporation it yields a gummy uncrystallizable mass.

ZINC ACETATE, $Zn(C_2H_3O_2)_2$.—This salt may be prepared by dissolving metallic zinc, zinc oxide, or zinc carbonate, in acetic acid, or by the decomposition of zinc sulphate by acetates of lime or lead, similar to the acetate of manganese; the decompositions in these instances being represented in the annexed equations:—

$$Zn + 2C_2H_4O_2 = Zn(C_2H_3O_2)_2 + 2H.$$
$$ZnO + 2C_2H_4O_2 = Zn(C_2H_3O_2)_2 + H_2O.$$
$$ZnCO_3 + 2C_2H_4O_2 = Zn(C_2H_3O_2)_2 + H_2O + CO_2.$$
$$ZnSO_4 + Pb(C_2H_3O_2)_2 = Zn(C_2H_3O_2)_2 + PbSO_4.$$

The acetate is obtained in the first three instances simply by evaporation, and in the latter, after agitating the mixture, filtering and evaporating the filtrate, the salt crystallizes in flexible, opalescent, six-sided tables, Fig. 38, which effloresce slightly in the air. According to SCHINDLER, the crystals contain 3 equivalents of water when deposited from cold solutions, but only 1 equivalent when they are formed from concentrated hot ones.

Fig. 38.



Technically, the best recipe is to dissolve 4 parts of the sulphate of zinc, and 7½ parts of acetate of lead, each in 3 parts of hot water, mixing the solutions, agitating, and after the sulphate of lead has deposited, drawing the clear liquid off to crystallize.

METALLIC ACETATES.—The acetates are characterized by the pungent odour of acetic acid which they emit when heated with sulphuric acid. When heated with lime they furnish acetone, which has a peculiar and unmistakable odour. When distilled with caustic potash they yield marsh gas (upon this reaction the best method of obtaining this gas is founded). Heated with arsenious oxide (As_2O_3), metallic acetates give rise to cacodyl (arsen-di-methyl, $As_2(CH_3)_6$, By this reaction extremely minute quantities of acetic acid can be detected.

PYROXYLIC SPIRIT OR WOOD NAPHTHA.—*Hydrate of Methyl*, *methylic alcohol*, *wood spirit*; *ésprit de bois*, French; *holzgeist*, *holzessiggeist*, German. Formula—

$$CH_4O = CH_3\,(OH) \text{ or } \left.\begin{matrix}CH_3\\H\end{matrix}\right\}O$$

This substance was first observed by TAYLOR in 1812, in the watery liquid obtained by the destructive distillation of wood, and its properties were subsequently more fully investigated by DUMAS and PELIGOT in 1835. Crude pyroligneous acid (wood vinegar) commonly contains about 1 per cent. of this alcohol, which is separated from the rest of the liquor by collecting apart the first portions which distil over. The acid solution thus obtained is neutralized with lime, and the clear liquor, when separated from the oil which floats on the top and the sediment which precipitates, is again distilled. The volatile liquid thus obtained is strengthened by rectification.

Pyroxylic spirit is a limpid, inflammable, colourless liquid, of a penetrating spirituous odour and a disagreeable burning taste. Its specific gravity is 0·8142 at 32° Fahr. (0° C.), 0·798 at 68° Fahr. (20° C.). Its boiling point varies according to the nature of the vessel, but may be taken as about 150° Fahr. (65°·5 C.). Wood spirit mixes with alcohol, ether, and water, and essential oils in all proportions; it dissolves most of the resins and gums, and is thus of considerable value in the arts, being used as a solvent in place of ordinary alcohol. It was formerly much used as a substitute for spirits of wine in spirit lamps. It burns with a very pale flame, producing in its combustion carbonic anhydride (CO_2) and water.

A solution of shellac and other resins in pyroxylic spirit is extensively used for stiffening the basis of silk hats. Furniture polishes are also made with it. The hydrates of the alkalies dissolve in wood spirit, but at the same time colour it brown. URE proposed this reaction as a means of detecting small quantities of wood spirit when mixed with alcohol, since the latter is unaffected by alkalies until the lapse of a considerable time; whereas when alcohol contains even as little as 2 per cent. of wood spirit the addition of powdered caustic potash causes it to acquire a yellow tint in ten minutes, and to become completely brown in half an hour.

This alcohol has never been produced by fermentation. BERTHELOT has obtained it artificially by acting on marsh gas with chlorine, and afterwards decomposing the chloride thus formed with aqueous potash. As marsh gas can be produced synthetically, it follows that the synthesis of wood spirit is also practicable.

Methylic alcohol is decomposed by passing through a red hot tube into acetylene and other substances. By exposure to the air, in contact with spongy platinum or platinum black, it is oxidized to formic acid thus:—

$$\overset{\text{Methylic Alcohol.}}{CH_4O} + O_2 = \overset{\text{Water.}}{H_2O} + \overset{\text{Formic Acid.}}{CH_2O_2}.$$

Bleaching powder (hypochlorite of calcium) converts wood spirit into chloroform ($CHCl_3$).

$$CH_4O + 4Cl = CHCl_3 + H_2O + HCl.$$

Potassium and sodium dissolves in it with evolution of hydrogen, forming potassium and sodium methylates (CH_3KO and CH_3NaO). Chlorine gas decomposes wood spirit with flame and detonation, forming chlorinated compounds together with hydrochloric acid. Concentrated sulphuric acid produces methyl sulphuric acid (CH_3HSO_4). This liquid, when heated together with wood spirit, yields dimethylic sulphate, $(CH_3)_2SO_4$.

In order to prepare commercial wood spirit, the distillates from the wood are received in a tank or other suitable vessel, and the whole allowed to rest for some time. The greater part of the tarry and other resinous and oily matters soon precipitates, the spirit and acid liquor forming a supernatant layer, which is drawn off into another tank by means of an overflow pipe or siphon. The liquids are sometimes made to percolate a filter of coarse gravel in their passage from the first tank, by which expedient a large quantity of the matters held in suspension by the acid liquor is separated.

The purification of the spirituous and acid products is effected in two ways—In the first the spirit is distilled directly from the crude liquor; in the second the acid is neutralized with milk of lime before being submitted to distillation to obtain the spirit.

In the first instance the liquor is pumped into copper stills of about 500 gallons. Heat is sometimes applied by means of coils of copper pipe placed in the interior of the stills, through which steam from an adjacent steam-boiler is forced; in other cases the heat is applied externally, either by steam-pipes coiling round the still, or by the direct application of fire. The distillation is continued till nearly all the spirit has passed over, which is known by throwing a portion of the distillate on a bright fire; if much spirit still remains the liquor inflames, giving a whitish light.

In most instances the whole of the naphtha is expelled when about 100 gallons are condensed in the receiver. When no more vapours are eliminated the receiver is changed, and the distillation of the acid liquor proceeded with till nearly all the liquid has passed over; the remaining tarry and oleaginous products are then run off into a tank through a stopcock in the bottom of the still. Should the acid be not immediately wanted, the remaining 400 gallons, after the expulsion of the spirit, are drawn off into the tank and allowed to rest, so as to deposit the excess of impurities, and afterwards pumped up to other vessels as required.

In the second method instanced the acid liquor is first neutralized by means of hydrate of lime, and the spirituous and crude acetate of lime liquors afterwards distilled in sheet-iron stills or boilers to obtain the naphtha. Fire is applied directly to the under part of the boiler. The remaining acetate of lime liquor is drawn off, and allowed to remain in tanks or reservoirs till required for use. The deposit of tarry matters which accumulates in the first receiver of the condensing apparatus, attached to the wood carbonizer, is also subjected to distillation, and the crude spirit therefrom added to the former portions.

The further purification of the crude spirit obtained in the preceding distillation, is effected by repeated rectifications, over at first caustic lime, and then over a mixture of lime and caustic potassa; and in order to detach the small quantity of ammonia that is formed, the final distillation of the product is carried on with a little sulphuric acid. In some factories the distillates are rectified over chalk, and in some others over chalk and bicarbonate of soda. Copper stills are invariably used, and the heat applied either by steam pipes coiled in the interior of the still, or the lower half of the retort is encased in an iron jacket, to which the fire is directly applied.

The spirit thus obtained is colourless, and varies in specific gravity from ·870 to ·832.

A few years ago WILDSMITH obtained a patent for the purification of spirituous and acid products of the distillation of wood. The principle of his invention is the application of an oxidizing agent, together with the influence of light, by which treatment he proposes to oxidize the hydrocarbons, which are unessential to the spirit and acid, but whose presence communicates a disagreeable odour to the products. Bichromate and permanganate of potassa are the salts he prefers, and which he employs in the following manner:—"The liquors are placed in a tank secluded from the air or the accidental admixture with dirt or other impurities, by means of a glass or glazed cover, so as to admit the light, which is necessary for the success of this process. Four ounces, avoirdupois, of the forementioned compounds, in fine powder, are used to every imperial gallon of the liquid; this may be thrown into the tank with the liquid, or the salt may be dropped into the solution gradually, and the whole well agitated. The time required to effect the purification of the spirit depends upon the quantity of tarry matters present; but in ordinary cases from fourteen to twenty-one days will suffice. Sunny weather favours the purification of the spirit and acid from the foreign compounds, as it brings about the combination of the oxygen of the oxidizing agent with the carbonaceous impurities in a very considerable measure. Other compounds, such as oxide opermanganese, or a properly regulated stream of chlorine gas passed through the solution, might answer the purpose equally well, but the permanganate and bichromate of potassa or soda offer the greatest facilities in their use; besides, they are, by effecting the intended purpose, converted into bodies which exert no diminishing action upon the substances sought, and which may be easily separated from the spirituous and acid liquor by distillation. When the purifying reagents have been added, the fluid should be agitated once or twice daily, in order to bring the solution of the salts into immediate contact with the impure liquor; during the intervals the cover should be kept on. Frames of glass or mica in the cover are made to slide backwards and forwards, in order to allow the liquid to be examined, and learn how far the purification has advanced. If it be desired to hasten the reaction, sulphuric or hydrochloric acid is added, in the proportion of 1 lb. of acid to 50 gallons of spirit. The object of these additions is to liberate the acid more freely from its combination with the potassa, and thus bring the whole of the oxygen within reach of the hydrocarbons and the other analogous impurities. When the oxidation of the impurities is completed, the liquor is distilled in the usual way, and a clear colourless product, without smell, and capable of being burned in lamps or used for any other required purpose, is obtained."

The general yield of pyroxylic spirit is from 1¼ gallon to 2½ gallons, and occasionally as much as 3 gallons, from each ton of wood. This variation in quantity is often due to the kind of wood carbonized, but more frequently to negligence in regulating the proper degree of heat.

The subjoined tabular view of the percentage amounts of spirit in naphthas of different specific gravities was drawn up by Dr. Ure. The spirit operated upon in these experiments was purified by distillation over quicklime, at the heat of a water-bath, the temperature being so regulated that the liquid had a density of ·8136 at 60° Fahr. (15° C.).

Sp. gr.	Real Spirit, per cent.	Over Excise proof.	Sp. gr.	Real Spirit, per cent.	Over Excise proof.
·8136	100·00	..	·9032	68·50	13·10
8216	98·00	64·10	9060	67·56	11·40
8256	96·11	61·10	9070	66·66	9·30
8320	94·34	58·00	9116	65·00	7·10
8384	92·22	55·50	9154	63·30	4·20
8418	90·90	52·50	9184	61·73	2·10
8470	89·30	49·70			Underproof.
8514	87·72	47·40	9218	60·24	0·60
8564	86·20	46·60	9242	58·82	2·50
8596	84·75	42·20	9266	57·73	4·00
8642	83·33	39·90	9296	56·18	7·00
8674	82·00	37·10	9344	53·70	11·00
8712	80·64	35·00	9386	51·54	15·30
8742	79·36	32·70	9414	50·00	17·80
8784	78·13	30·00	9448	47·62	20·80
8820	77·00	27·90	9484	46·00	25·10
8842	75·76	26·00	9518	43·48	28·80
8876	74·63	24·30	9540	41·66	31·90
8918	73·53	22·20	9564	40·00	34·20
8930	72·46	20·60	9584	38·46	35·60
8950	71·43	18·30	9600	37·11	38·10
8984	70·42	16·16	9620	35·71	40·60
9008	69·44	15·30			

Deville has likewise completed a table, showing the quantity of real spirit in naphtha of different specific gravities at 50° Fahr. (10° C.), which differs somewhat from that of Ure. Deville considers that Ure's spirit contained some water.

Methylic Alcohol.	Specific gravity.
100 per cent.	0·8070
90 “	0·8371
80 “	0 8649
70 “	0·8873
60 “	0·9072
50 “	0·9232
40 “	0 9429
30 “	0·9576
20 “	0·9709
10 “	0·9751
5 “	0·9857

The same authority says, if the above results are brought to a temperature of 60° Fahr. it will be found there is an almost complete correspondence between alcohol and wood spirit, and that the latter, equally with the former, exhibits a maximum of contraction, which always occurs when one part of wood spirit is combined with three parts of water; that is, in a mixture containing 45·75 per cent. of water. The lowest specific gravity to which pyroxylic acid has been brought in this country is 0·812. Dumas, however, states that its density at the temperature of 68° (20° C.) is 0·798, and that of its vapour 1·120, and that its point of ebullition is 152° (66·5° C.) at a pressure of 30 inches. The true specific gravity appears to be 0·798, as found by Dumas and Mitscherlich; and from several experiments performed by Muspratt its boiling point ranges between 150° and 160° Fahr. (65°·5 and 71° C.).

Acetone, methyl acetate, dimethyl acetate, and the empyreumatic oils, being in variable proportion in wood naphtha, cause its density to alter considerably; its solvent power, as regards shell-lac, sandarac, and other resins, is likewise materially affected by the presence or absence of such bodies. Their existence is attributed to the various methods of preparing the spirit, and also to the want of proper care in managing the operations to which it is subjected. Pure pyroxylic spirit will not answer every purpose for which it is required in the arts. The purest, and that possessing the lowest specific gravity, is preferable for lamps as a source of heat; but for dissolving resins, and especially gum sandarac and mastic, painters and French polishers choose spirit holding some of the essential oils in solution. Wood spirit of a low specific gravity, and free from acetone, is procured by liming the raw liquor and distilling; whilst the best menstruum for resins is obtained by distilling off the refined portion of the crude liquor without saturating with the caustic earth. The spirit in the former case has an incipient gravity and is miscible with water, while in the latter it is weaker, and water renders it milky.

To procure pure pyroxylic acid, the crude commercial spirit is saturated with fused chloride of calcium, and heated on the water bath as long as volatilization continues. The chloride of calcium acts by combining with the methylic alcohol to form a solid crystalline compound, whilst the other substances present separate as an oily liquid, which is poured off; the last traces of acetone, methyl acetate, &c., are removed by heating the solid mass to 212° Fahr. (100° C.), at which temperature the calcium compound is not decomposed. On adding water, however, and distilling, the dry residue yields methyl alcohol, which is dried over quick lime and again distilled over a water bath. The alcohol thus obtained is not perfectly pure. To prepare the pure compound, methylic ether must first be produced. Purified wood spirit is mixed with its own weight of sulphuric acid and twice its weight of potassium binoxalate, and the whole distilled. As soon as crystals of methyl oxalate appear in the neck of the retort the receiver is changed, the distillation being continued as long as the ether comes over. The crystals are powdered and pressed between filter paper. On distilling this pure ether with water, methylic alcohol passes over and oxalic acid remains; thus:—

Methyl Oxalate. Methylic Alcohol. Oxalic Acid

$$\underbrace{(CH_3)_2C_2O_4}_{\text{Methyl Oxalate.}} + 2H_2O = \underbrace{2CH_4O}_{\text{Methylic Alcohol.}} + \underbrace{C_2H_2O_4}_{\text{Oxalic Acid}}.$$

The aqueous methylic alcohol is redistilled from a water bath, dried over quicklime, and then rectified.

Pure pyroxylic spirit is readily distinguished from acetone by adding to it a concentrated solution of chloride of calcium, which is miscible with the wood spirit, but not with the pyroacetic spirit, or acetone.

Methylated Spirit.—Common alcohol is used in so many manufactures as a solvent, that the duty on spirits was found to be a serious detriment to trade. The excise regulations, however, now permit the sale of a mixture of 10 parts of crude methylic alcohol (pyroxylic spirit) and 90 parts of absolute alcohol, duty free. This mixture, termed "methylated spirit," is suitable for all the ordinary purposes to which spirit of wine is applied in the manufactures, but cannot be used for making gin or other spirituous drinks, on account of its extremely offensive odour and taste. The methylic alcohol cannot be separated by distillation. Of late years methylated spirit has been used in nearly all the manufacturing processes in which pyroxylic spirit was formerly employed.

Acetic Ether.—*Acetate of Ethyl, Ethylic Acetate; essigäther, essignaptha, essigsaures æthyloxyd*, German; *ether acétique*, French; $C_2H_3O_2,C_2H_5$ or $C_4H_3O_2$.

This body is the most important of the numerous compounds termed generically acetic ethers.

It is formed by the substitution of the radicle of ethyl alcohol (spirits of wine, "alcohol" *par excellence*) for the basic hydrogen of acetic acid, thus:—

$$\begin{aligned}\text{Acetic acid} &= C_2H_3O_2 + H.\\ \text{Ethyl alcohol} &= C_2H_5 \quad + HO.\end{aligned}$$

$$\underset{\text{Acetic acid.}}{(C_2H_3O_2 + H)} + \underset{\text{Ethyl alcohol.}}{(C_2H_5 + OH)} = \underset{\text{Ethyl acetate.}}{C_2H_3O_2,C_2H_5)} + \underset{\text{Water}}{H_2O.}$$

The radicle of any monatomic alcohol may in like manner replace the loosely attached hydrogen atom, and in this way are formed acetate of methyl ($C_2H_3O_2,CH_3$), acetate of butyl ($C_2H_3O_2,C_4H_9$), acetate of amyl ($C_2H_3O_2,C_5H_{11}$), and other acetates of monatomic alcohols. In like manner the radicle of benzyl alcohol, $C_7H_8O = C_7H_7(OH)$, forms benzyl acetate, $C_2H_3O_2,C_7H_7$:—

$$\underset{\text{Acetic acid.}}{(C_2H_3O_2,H)} + \underset{\text{Benzyl alcohol.}}{(C_7H_7,OH)} = \underset{\text{Benzyl acetate.}}{C_2H_3O_2,C_7H_7} + \underset{\text{Water.}}{H_2O.}$$

Ethyl acetate was discovered by Lauraguais in 1759. This chemist obtained it by heating alcohol with acetic acid. It is readily prepared by distilling to dryness 3 parts of potassium acetate with 2 parts of absolute alcohol; or 10 parts of sodium acetate, 6 parts of alcohol, and 15 parts of sulphuric acid; or 16 parts of dry sugar of lead, with 6 parts of sulphuric acid, spec. grav. 1·84, and 4½ parts of water. Heating must at first be conducted with great caution; as the distillation proceeds the temperature may be raised.

The fluid which passes over is first mixed with a weak solution of carbonate of soda, or some other alkali, till neutralized, and then the supernatant layer of aqueous acetic ether is drawn off and agitated repeatedly with dry potassium carbonate, until that salt is no longer moistened with it; the solution is then distilled to procure the pure ether. Chloride of calcium should not be used, since it is soluble in acetic ether, and gives rise to subsalts.

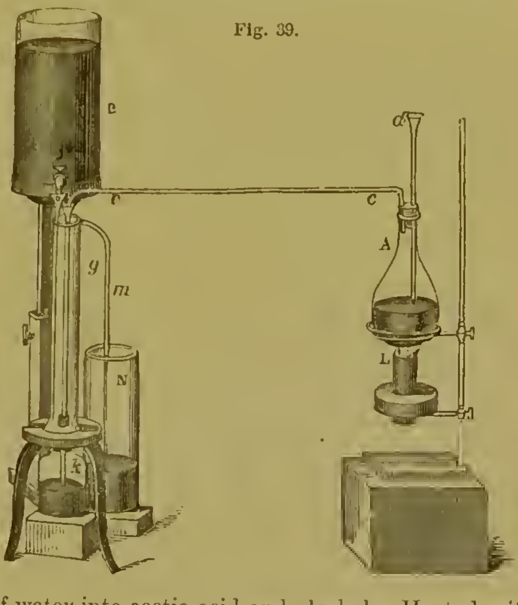
Acetic ether is a colourless mobile liquid, having an agreeable refreshing etherial odour and a pleasant taste; it burns with a yellowish flame, producing acid vapours. Its spec. grav. is 0·89 at 60° Fahr. (15° C.), 0·9146 at 32° Fahr. (0° C.).—Kopp. It boils at 165° Fahr. (74° C.), and is converted into vapour of spec. grav. 3·067.

Acetic ether is soluble in 11 to 12 parts of water, and in alcohol and ether in all proportions.

Fig. 39 is a convenient form of apparatus for making acetic ether. A is a large flask, furnished with an air-tight cork, perforated with two holes, through one of which a safety or funnel tube, *d*, passes, and the other receives the tube, *c c*, bent at right angles as seen in the figure, through which the vapours pass to the condenser. The condenser, *g*, is a wide cylinder, having an aperture at the bottom, through which the bent end of the tube passes into a flask, *k*, placed beneath the cylinder to receive the distilled products. A cistern, E, supplies the cylinder, *g*, by the stopcock, *j*, with cold water, which is conducted to the bottom by the funnel pipe, *o*, and the partly-heated water passes off by the overflow pipe, *m*, into the vessel, N. Heat is applied to the flask by means of the spirit lamp, L, or a Bunsen burner.

Acetic ether is unalterable in dry air; in a moist atmosphere it is after a time converted by absorption of water into acetic acid and alcohol. Heated with a caustic alkali, acetic ether splits up in like manner, an acetate and alcoholate being formed, thus:—

Fig. 39.



$$\underset{\text{Acetic ether.}}{2(C_2H_3O_2,C_2H_5)} + \underset{\text{Baryta.}}{2(BaO)} = \underset{\text{Barium acetate.}}{Ba(C_2H_3O_2)_2} + \underset{\text{Barium alcoholate.}}{BaO_2(C_2H_5)_2.}$$

Acetate of lead is the salt commonly employed when making acetic ether on a large scale; the proportions used being those given above. The sulphuric acid and alcohol are mixed in a vessel surrounded by ice, and the mixture, when cooled, is poured upon the finely-divided lead salt in the flask; the apparatus is adjusted, and a gentle heat applied at first, which is gradually increased towards the end of the distillation. The condenser is well cooled, and the receiver may be advantageously immersed in ice-cold water. The acetic ether obtained is rectified as before-mentioned.

When this ether is poured upon chloride of calcium, combination takes place, and a crystalline mass results, from which, by the addition of a small quantity of water acetic ether again separates. By digestion in a solution of potassa acetic ether suffers complete decomposition; acetic acid unites with the alkali, and ether passes off: the same effect is produced when the ether is distilled with lime. Heated with strong sulphuric acid, acetic ether is converted into oxide of ethyl (common "ether," $(C_2H_5)_2O = C_4H_{10}O$) and acetic acid. Hydrochloric acid converts it into chloride of ethyl (C_2H_5Cl) and acetic acid. Pure acetic ether does not react on blue litmus paper, nor should it be coloured by sulphuretted hydrogen. It dissolves resins, sulphur, phosphorus, &c., like ether.

This compound is a constituent of many wines; it is almost always present in wine vinegar, and is used as a constituent of several of the pharmaceutical preparations employed in medicine. From its power of dissolving resins and essential oils, it can be advantageously applied in the preparation of varnishes.

ACETATE OF METHYL, *Methylic Acetate*, *Pyroacetic Spirit; essigsaures holzæther*, German; *æther lignosus* — $C_2H_3O_2,CH_3$ or $C_3H_6O_2$. This liquid is one of the constituents of crude wood vinegar. It was first noticed by DUMAS and PÉLIGOT in 1835. These chemists prepared it by distilling 2 parts of pyroxylic spirit with 1 part of glacial acetic acid and 1 part of sulphuric acid, and agitating the distillate with chloride of calcium. After allowing the mixture to settle the supernatant liquid is methylic acetate, contaminated with sulphurous acid and wood spirit. The former impurity is removed by agitation with quicklime, and the latter by leaving the methylic acetate in contact with chloride of calcium for twenty-four hours.

WEIDMANN and SCHWEIZER, who first detected this compound in wood vinegar, prepare it by distilling a mixture of 2 parts pyroxylic spirit, 1 part of acetic acid, and 1 part of sulphuric acid; or 3 parts of wood spirit, 14½ parts of dry lead diacetate (sugar of lead), and 5 parts of sulphuric acid; or, and this is the mode they prefer, 1 part of potassium acetate and 2 parts of sulphuric acid. The distillation is arrested immediately sulphurous acid begins to be evolved, the excess of wood spirit is removed by chloride of calcium, and the liquid distilled from soda carbonate; further purification is effected as in the case of ethylic acetate (acetic ether *q. v.*).

Methylic acetate is a colourless liquid, having an odour much resembling that of acetic ether; its spec. grav. at 32° Fahr. (0° C.) is 0·9562, at 70° Fahr. (21° C.) 0·919.—KOPP. Vapour density, 2·563.—DUMAS and PÉLIGOT.

Acetate of methyl is soluble in water, and mixes with alcohol, ether, and pyroxylic spirit in all proportions. Its aqueous solution is but slightly decomposed on boiling. Alkalies transform it into methylic alcohol and an acetate. Soda lime decomposes it with much violence into a sodium acetate and formiate, with evolution of hydrogen. Sulphuric acid splits it up into acetic acid and methyl sulphuric acid. much heat being given off during the reaction.

ACETONE, *Pyroacetic Spirit*, *esprit pyroacétique*, of the French; *essiggeist*, *brenzessiggeist*, German— $C_3H_6O = C_2H_3O,CH_3$. The ketone of acetic acid. It is derived from acetic aldehyde by the substitution of the alcohol radicle CH_3, for the hydrogen in the group COH.—GERHARDT and WILLIAMSON.

Acetic aldehyde. — Acetic ketone or acetone.

$$CH_3,CO(H). \qquad CH_3,CO(CH_3).$$

This compound was first observed by COURTENVAUX in 1754. The brothers DEROSNE subsequently investigated its properties, and named it pyroacetic ether. CHENEVIX, however, concluded from experiment that it was not an ether, and termed it pyroacetic spirit. Its composition was definitely ascertained by DUMAS and LIEBIG.

KANE looked upon acetone as mesitylic alcohol, C_3H_5,HO (C_3H_5 being the radicle mesityl); but his views were proved to be erroneous.

This body occurs in the destructive distillation of all acetates. When required it is prepared by distilling barium or calcium acetate; acetone is given off, and the carbonate of the alkaline metal remains.

Calcium diacetate. — Acetone. — Calcium carbonate.

$$Ca(C_2H_3O_2)_2 = C_3H_6O + CaCO_3.$$

When the alkaline acetate is pure the product is pure acetone. It can also be made by distilling in an iron retort a mixture of 2 parts of sugar of lead (lead diacetate) and 1 part of powdered quicklime. The product must in this case be saturated with potassium carbonate, rectified over chloride of calcium several times, and finally distilled by the heat of a water bath. The condenser must be kept carefully cooled, and only those products collected which pass over at 140° Fahr. (60° C.).

A large quantity of acetone is obtained as a bye product in the manufacture of aniline, when distilling the mixture of acetate of iron and aniline, produced by the action of iron and acetic acid upon nitrobenzene.

Acetone is likewise formed during the dry distillation, together with lime, of sugar, tartaric acid, gum arabic, and like substances, and also by the oxidation of citric acid, by heating it with permanganate of potassium or a mixture of binoxide of manganese and dilute sulphuric acid. — PEAN DE SAINT GILLES.

Citric acid. — Acetone. — Carbonic acid. — Water.

$$C_6H_8O_7 + O = C_3H_6O + 3CO_2 + H_2O.$$

When acetic acid is decomposed by being passed through a porcelain tube heated to redness, acetone is one of the products—

Acetic acid. — Acetone. — Carbonic acid. — Water.

$$2C_2H_4O_2 = C_3H_6O + CO_2 + H_2O.$$

Acetone is a colourless limpid liquid, having a very agreeable ethereal odour and a burning taste. Its specific gravity is at 65° Fahr. (18° C.) 0·7921, according to LIEBIG; at 32° Fahr. (0° C.) 0·814.—KOPP. Its vapour density is 2·0025.—DUMAS. Its boiling point is 130° Fahr. (56° C.).—DUMAS. It does not solidify at 5° Fahr. (— 15° C.). Acetone is very inflammable, and burns with a bright yellow flame. It is soluble in all proportions in ether, alcohol, and in water. It is a solvent for nearly all gums and resins, camphors and fats. Gun cotton dissolves in it with great facility.

LIMPRICHT has formed definite compounds of acetone with the alkaline bisulphites. The solution of bisulphite should be very concentrated; the action then takes place somewhat rapidly, and with evolution of heat.

Acetone vapour is decomposed at a red heat into charcoal and *dumasine*, a peculiar empyreumatic oil. Passed over potassium hydrate at a red heat it forms marsh gas and carbonic acid—

$$\begin{array}{ccccccc} \text{Acetone.} & & \text{Potassium hydrate.} & & \text{Potassium carbonate.} & & \text{Marsh gas.} \\ C_3H_6O & + & 2KHO & = & K_2CO_3 & + & 2CH_4; \end{array}$$

or at a lower temperature the products are as below—

$$\begin{array}{ccccccccccc} \text{Acetone.} & & \text{Potassium hydrate.} & & \text{Water.} & & \text{Potassium acetate.} & & \text{Potassium formate.} & & \text{Hydrogen.} \\ C_3H_6O & + & 2KHO & + & H_2O & = & KC_2H_3O_2 & + & KCHO_2 & + & 6H. \end{array}$$

ALCOHOL.—*Hydrate of Ethyl; Ethyl alcohol, Hydroxyl ethane, Methyl carbinol.—Alcool*, French; *weingeist*, German; *spiritus rectificatus*, Pharmacopœia.

$$C_2H_6O = C_2H_5,HO = \left\{\begin{matrix} CH_3 \\ CH_2 \\ HO \end{matrix}\right. = C \left\{\begin{matrix} CH_3 \\ H \\ H \\ HO \end{matrix}\right. \text{ or}$$

hydrated ethene (ethylene) C_2H_4,H_2O.

The word alcohol, derived from the Arabic *al kohol*, signifying a trituration or grinding, was at first applied to the preparation of antimony used by oriental women for painting their eyebrows. The appellation was afterwards given to other fine powders, and to highly-rectified spirits, but for what reason is not very clear.

There is no evidence of the ancients being acquainted with alcohol or ardent spirits; in fact, there is every reason to believe the contrary, and that even distillation was quite unknown to them. The method followed by DIOSCORIDES to obtain quicksilver from cinnabar—sulphide of mercury—was really a process of distillation: he mixed the cinnabar with iron filings, put the mixture into a pot, to the top of which a cover of stoneware was luted; heat was applied, and when the process was terminated, the mercury was found adhering to the side of the cover. DIOSCORIDES possessed much penetration and judgment, but he was unacquainted with the method of adapting a receiver to his pot, or he would not have proceeded as above recounted. Another strong corroboration of the fact, that alcohol was unknown in former times, is that neither the poets, historians, naturalists, nor medical men, make the slightest allusion to ardent spirits.

This compound, though viewed in many different aspects, as its varying formulæ denote, may be regarded as an atom of water (H_2O) in which half the hydrogen is replaced by ethyl (C_2H_5), *i.e.*, a compound of the radicle hydroxyl (HO) with the radicle ethyl ($C_2H_5 + HO = C_2H_6O$).

In the strict chemical sense, the term *alcohol* is employed as a generic name for a class of bodies belonging to the same type, or framed upon the same principle. Just as *salt*, originally applied to chloride of sodium, or the condiment with which we season our food, is now extended to a class of bodies often destitute of any savour; as *metal* designates several substances which do not possess either the malleability, the specific gravity, or the power of resisting heat which characterized those from which the term was borrowed; or as *acid* appertains to many compounds having neither a sour taste nor any caustic properties; so *alcohol* indicates a class, some members of which, far from being volatile, are not even liquid, and instead of igniting easily, require an elevated temperature for kindling.

All neutral compounds of carbon, hydrogen, and oxygen which react directly upon acids, so that water is eliminated and ethers are produced, are now termed alcohols.

Alcohols are formed from hydrocarbons by the substitution of one equivalent of the radical hydroxyl for one atom of hydrogen, in the case of monatomic alcohols, of two equivalents in the case of diatomic, three equivalents for triatomic alcohols, &c. These are generally spoken of as monatomic and polyatomic alcohols.

The chief monatomic alcohols are the following:—From methane or marsh-gas (CH_4) methyl alcohol is constituted thus: CHHH(HO)—

		Termed by KOLBE.	
Methyl alcohol,....................	CH_4O = CHHH(OH)	Carbinol,............	$C(OH)H_3$.
Ethyl alcohol,....................	$C_2H_6O = CHH(CH_3)(OH)$	Methyl carbinol, ...	$C(OH)H_2(CH_3)$.
Propyl alcohol,..................	$C_3H_8O = CHH(C_2H_5)(OH)$..........	Ethyl carbinol,......	$C(OH)H_2(C_2H_5)$.
Butyl or quartyl alcohol,.........	$C_4H_{10}O = CHH(C_3H_7(OH)$	Propyl carbinol,.....	$C(OH)H_2(C_3H_7)$.
Amyl or quintyl alcohol,.........	$C_5H_{12}O = CHH(C_4H_9)(OH)$.		
Sextyl alcohol, ...	$C_6H_{14}O = CHH(C_5H_{11})(OH)$.		
Septyl alcohol, ...	$C_7H_{16}O = CHH(C_6H_{13})(OH)$.		
Octyl alcohol, ...	$C_8H_{18}O = CHH(C_7H_{15})(OH)$.		
Nonyl alcohol, ...	$C_9H_{20}O = CHH(C_8H_{17})(OH)$.		

Alcohols are divided into groups, which are termed respectively primary, secondary, or tertiary, according as the carbon atom combining with the atom of hydroxyl is in combination with one, two, or three other carbon atoms.

KOLBE's nomenclature is convenient for the expression of the isomeric alcohols formed by the substitution of atoms of methyl, ethyl, propyl, &c., for the hydrogen atom in the group, which he terms carbinol (methyl alcohol). In this way we have:—

Dimethyl carbinol (isopropyl alcohol), $C(OH)H(CH_3)_2$
Isopropyl carbinol (isoquartyl alcohol) $C(OH)H_2CH(CH_3)_2$
Methyl-ethyl carbinol (secondary quartyl alcohol), $C(OH)H(CH_3)(C_2H_5)$
Trimethyl carbinol (tertiary quartyl alcohol) $C(OH)(CH_3)_3$

Methyl alcohol has already been considered. (See Acetic Acid.)

Ethyl alcohol is the most important of all these compounds. It was known to the alchemists. Its discovery is said to be due to Albucasis and Arnold de Villeneuve, in the twelfth century. Raymond Lully, about the fourteenth century, was acquainted with spirits of wine, which he called *aqua ardens*, and made by distilling wine and concentrating the distillate by means of potassium carbonate. Lœwitz and Richter subsequently succeeded in completely separating the water by means of quicklime. Alcohol was first analyzed by De Saussure, who separated it into 100 parts by weight of olefiant gas and 63·58 of water: about equal volumes of olefiant gas (ethane, C_2H_4) and aqueous vapour (H_2O). The vapour density of alcohol, as given by Gay Lussac, agrees with this composition.

In a general and practical sense, by *alcohol* is ununderstood the pure spirit obtained by the distillation of liquids which have undergone vinous fermentation. It is the intoxicating principle of all vinous and spirituous liquors, as wine, brandy, whiskey, &c. Distilled from wine, it bears the name *spirit of wine*.

After the completion of fermentation distillation of the fermented body affords it either in a more or less concentrated state. Mere distillation, however, will not yield absolute alcohol, that is, alcohol free from water. For though absolute alcohol boils at 173° Fahr. (78°·4 C.), it has a great affinity for water, and dilution by water contracts its volume and raises its boiling point, so that the aqueous vapour corresponding to this temperature passes over with the spirit. At the lowest temperature at which the distillate is drawn off some aqueous vapours rise with the alcohol, and both are condensed simultaneously in the receiver; therefore, whatever the heat may be, the resulting alcohol is not anhydrous, but exhibits a density of 0·820 to 0·830.

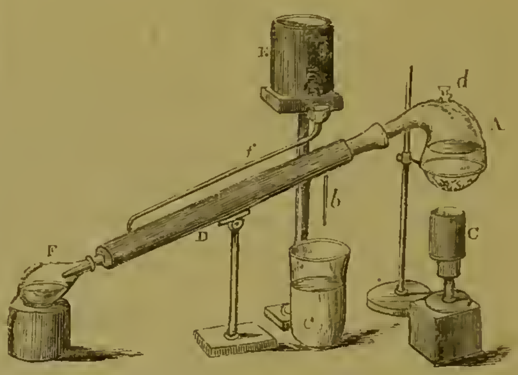
To procure absolute alcohol is a difficult and tedious operation: spirit of wine has to be distilled at a moderate heat from some hygrometric substance, such as anhydrous copper sulphate, calcium chloride, potassium carbonate, or quicklime. The best adapted for the purpose is quicklime reduced to coarse powder and put into a retort with the alcohol, and the whole mixed by agitation; the neck of the retort is then securely corked, and the mixture left for several days, during which period the water unites with the quicklime and converts it into calcium hydrate (CaH_2O_2), leaving the anhydrous spirit, which may be distilled off from the mixture by the heat of a water-bath.

Fig. 1 is the apparatus usually employed in the laboratory for this purpose: A is a glass retort; heat is applied by the lamp, C; D is a Liebig's condenser, through which the tube receiving the beak of the retort passes to the flask, F. Cold water from the tank, E, enters the condenser, D, by the funnel tube, *f*, and the heated water is discharged by *b* into the vessel, *c*. The mouth of the receiver is closed by a perforated cork, to prevent the access of air and the absorption of aqueous vapours. The dilute alcohol and coarsely-powdered dehydrating substance are introduced through the stoppered opening, *d*, the stopper replaced, and the distillation carried on at as low a temperature as possible—173° to 176° Fahr. (78° to 80° C.). Repeated distillations with fresh portions of the hygroscopic substance used are necessary to free the alcohol completely from water.

To enable the quicklime to abstract all the water it should be left in contact with the alcohol for at least three or four days at the temperature of 95° Fahr. (35° C.): 1 part of 91 per cent. alcohol requires somewhat more than 1 part of quicklime in order to yield alcohol of 99·2 per cent. by filtration, and absolute alcohol by slow distillation. Alcohol of 94 per cent. requires only $\frac{5}{8}$ part of lime, and alcohol of 97 per cent. only $\frac{5}{16}$ of a part.

Mendelejeff states that to obtain the alcohol absolute, spirit of not more than ·792 specific gravity must be taken, and its digestion over the quicklime continued for not less than two days, or else for a few hours at between 122° to 140° Fahr. (50° to 60° C.). Even then, on distilling, he found only the middle portions anhydrous.

Fig 1.



By boiling the spirit with the lime in a vessel fitted with an inverted condenser for about an hour, and then distilling, the whole product is obtained anhydrous. If the spirit contains more than 5 per cent. of water it is necessary to repeat the treatment with lime two or more times. But with weak spirit care must be taken at first not to fill more than half the space occupied by the spirit with lime, as otherwise the vessel may be broken by the slaking of the lime. In this way several litres of spirit may be converted into absolute alcohol in a few hours.

A singular mode of concentrating alcohol has been proposed by Soemmering. He used an ox or a calf bladder, which was soaked for some time in water, and freed from the fat and the attached vessels. After being again inflated and dried, it was coated, the outside twice and the inside four times, with a solution of isinglass, and then charged with diluted spirit, leaving a small space vacant. On exposing it to heat the water evaporates, while the alcohol remains. The bladders can be repeatedly used. It is stated that by this means, with proper attention to the degree of heat applied, absolute alcohol may be obtained in from six to twelve hours. Soemmering states that he placed two bladders of the same size, having eight ounces of water in one and eight ounces of absolute alcohol in the other, over a sand-bath, where equal heat was communicated to both; after the lapse of four days all the water was evaporated and only one ounce of the alcohol. Smugglers, who carry spirits in bladders hid about their persons, have often said that although the liquor decreased in bulk yet it increased in strength.

Donovan, Stanford, and others, have repeated these experiments without being able to obtain these results to the extent specified, if, indeed, at all. The alcohol was used of various strengths, with small and large bladders, with thick ones and thin ones; but a diminution of strength was the invariable result, unless the spirit was very weak, when a very slight increase of strength took place.

Graham repeated Soemmering's experiment with better success. He found that when a jar filled with alcohol was covered with a dry membrane the alcohol escaped first; but when a moist bladder was completely filled with dilute alcohol the liquid decreased in bulk, and the water passed through the membrane, leaving a much larger percentage of alcohol in the bladder. Graham believed that Soemmering's experiment was an instance of arrested diffusion when more that 5 per cent. of alcohol was present; the action having some resemblance to the separating and secreting power of cells in the living organism.

Graham proposed to concentrate alcohol in the following manner:—A large shallow basin is covered to a small depth with recently-burned lime, in coarse powder, and a smaller basin, containing three of four ounces of commercial alcohol, is made to rest upon the lime; the whole is placed under the receiver of an air-pump, and exhaustion continued till the alcohol evinces signs of ebullition. Of the mingled vapours of alcohol and water which now fill the receiver, the quicklime is capable of uniting with the aqueous only, which are, therefore, rapidly withdrawn, while the alcohol vapour is unaffected; and as water cannot remain in the alcohol as long as the surrounding atmosphere is devoid of moisture, more aqueous vapour rises, which is likewise abstracted by the lime, and thus the process goes on till the whole of the water in the alcohol is removed. Several days are always required for this purpose, and in winter a longer time than in summer. On submitting alcohol of spec. grav. 0·827 to dehydration by this method, the following are the notes of the decreasing specific gravity after every twenty-four hours:—

When the liquid was introduced, the density was	0·827
Density after twenty-four hours' exposure to the action of the caustic lime, ...	0·817
Do. after second twenty-four hours,........	0·808
Do. after third do.	0·802
Do. after fourth do.	0·798
Do. after fifth do.	0·796

The longer time required for the concentration of the spirit in the winter season is seen from the following table, where the liquid, before inclosing it under the air-pump with the lime, had a spec. grav. 0·825:—

On determining the specific gravity after twenty-four hours, the result was, ...	0·817
Do. after the second twenty-four hours, ...	0·809
Do. after the third do.	0·804
Do. after the fourth do.	0·799
Do. after the fifth do.	0·797
Do. after the sixth do.	0·796

Quicklime possesses the property of uniting with a portion of alcohol vapour, and for this reason should not be used in great excess. Graham found that when four ounces of ordinary alcohol were exposed to the action of the lime, under the bell-jar of the air-pump, one-sixth of the alcohol was lost, on account of the absorption by the quicklime. Liebig states that in dehydration by lime half this alcohol is retained by the lime, but can be recovered by distillation. Hence the quantity of lime used should never exceed three times the weight of the alcohol, otherwise the loss of alcohol will become appreciable; the lime should be spread to as great an extent within the receiver as possible, that a larger absorbing surface may be presented to the vapour.

Connell states that if the lime be renewed every week the alcohol continues to lose water, becoming in four weeks of the density 0·795 at 60° Fahr. (15°·5 C.), and in twelve weeks becomes absolute alcohol; spec. grav. 0·7938.

Alcohol may also be concentrated by exposing it with lime in a close vessel. It cannot be concentrated over strong sulphuric acid, for this acid absorbs alcohol vapour with the same affinity with which it unites with water, the heat produced being very great; ethionic acid (C_2H_6,S_2O_7), a bibasic acid, is formed by this reaction. Calcium chloride is not well adapted for concentrating alcohol, on account of the affinity existing between these two bodies; since, if employed in excess, it forms a compound with alcohol which is not decomposed till a temperature is reached at which the alcohol is split up into olefiant gas (C_2H_4) and water; also, when submitted to distillation, the aqueous vapour absorbed by the chloride of calcium is apt to go over with the last portions of the alcohol, through the salt settling at the bottom of the retort and becoming over heated.

An excellent method for the preparation of pure alcohol was devised by Poggendorff.—Dissolve as much caustic potash in alcohol as it will take up, then add half its volume of water, and distil at a low temperature; a perfectly pure product is the result.

Drinkwater, in his investigation on the preparation of absolute alcohol and the composition of *proof spirit*, procured alcohol of the lowest specific gravity hitherto obtained. His mode of procedure was as follows:—Potassium carbonate was exposed to a red heat, to deprive it of water, and when sufficiently cool was pulverized and added to ordinary alcohol, of spec. grav. ·850, at 60° Fahr. (15°·5 C.), till it ceased to dissolve any more; the menstruum was then allowed to digest twenty-four hours, being frequently agitated, and the alcohol carefully separated by decantation. As much fresh-burned quicklime as was considered sufficient, when powdered, to absorb the whole of the alcohol, was introduced into a retort, and the alcohol added to it; after digesting forty-eight hours, it was slowly distilled into a water-bath at a temperature of about 180° Fahr. (82° C.).

The alcohol thus obtained was carefully redistilled, the retort again filled with fresh-burned and pulverized quicklime, and the same alcohol mixed with it; the mixture was then allowed to digest a whole week, at the ordinary temperature of the laboratory.

After this lapse of time, the alcohol was distilled off as before, and the distillate submitted to a second operation, which was conducted at first at the rate of about one drop in ten seconds, the heat of the water-bath being 165° Fahr. (74° C.). The distillation was continued thus till about one-twentieth of the whole had passed over, the object being to allow any minute quantity of water which the alcohol might still retain, to evaporate or diffuse itself into the atmosphere of absolute alcohol above it; the process was then continued rapidly, the heat of the bath being increased to 180° Fahr. (82° C.), till about one-twentieth more had passed over; the receiver was then changed, and the latter portions slowly eliminated. The specific gravity of the alcohol, taken twice, was ·7944, at 60° Fahr. This alcohol was again digested at a temperature of about 150° Fahr. (65°·5 C.) for fourteen days with quicklime previously heated to redness, as in the former experiment; it was then slowly distilled, out of contact with the external atmosphere, by means of a tube which passed from the condenser through a cork into the bottle in which it was to remain—the temperature at which it was distilled being 175° Fahr. (79°·3 C.)—and the first tenth part put aside as possibly containing a minute quantity of water; the remainder was then distilled off at 178° to 180° Fahr. (80°·8 to 82° C.). This alcohol was quickly transferred to a dry retort, and redistilled in a similar way, at a water-bath heat of 172° Fahr. (77°·6 C.); the first tenth part was set aside, and the remainder kept as pure anhydrous alcohol, or as free from water as it is possible to obtain it by this process. The specific gravity was then taken, the alcohol being kept, during the time of transference, as much as possible out of contact with the air. The results of four trials were as annexed:—

Temp. of room, 60° Fahr.	Barometer, 29·810 inches.
I.,	·793836
II.,	·793806
III.,	·793798
IV.,	·793804
Mean,	·793811

A portion of this alcohol was subsequently digested with quicklime for three months; it was then distilled, and its specific gravity was found to be exactly the same as before.

According to Kopp the specific gravity of alcohol is at 32° Fahr. (0° C.) 0·8095, at 60° Fahr. (15°·5 C.) 0·7939, at 68° Fahr. (20° C.) 0·792. The boiling point of alcohol is 173·1 Fahr. (78·4 C.) under the pressure of 760 mm.—Gay Lussac. The same chemist places its vapour density at 1·613 as compared with air, and 23·27 as compared with hydrogen.

Properties of Alcohol.—Pure anhydrous, or absolute alcohol, is a limpid colourless liquid, of a greater fluidity than water, having a penetrating but agreeable odour, and a hot pungent taste, owing to its abstracting water from the tissue of the tongue. When undiluted it exercises a poisonous action on the system, since it destroys the vital functions of the tissues by abstracting their constitutional moisture, by reason of its own great affinity for water. These violent effects are not produced when alcohol is taken in small quantities in a diluted state. It is a powerful stimulant and antiseptic.

Alcohol, when anhydrous, burns with a whitish flame, which deposits carbon on a cold surface held in it; when it is mixed with water the flame is quite blue, and no deposit of carbon is formed. In the combustion of alcohol very little light is emitted, but intense heat is given off: Boerhaave first showed that when its products of combustion are collected in proper vessels, water constitutes a large part of them.

Alcohol is exceedingly volatile: if a few drops be introduced into a jar of oxygen gas, it is readily converted into vapour, and a very explosive mixture is produced. When one volume of alcoholic vapour and three of oxygen are mixed, and ignited by the electric spark, a violent explosion ensues, and two volumes of carbonic acid and three volumes of aqueous vapour are formed.

When the sparks from an induction coil are passed through liquid alcohol it soon acquires an acid reaction, deposits black flocks, gives off gas, and forms a resinous substance. Addition of a small quantity of potassium hydrate facilitates the reaction. The evolved gases are similar in composition to that which is produced by the decomposition of alcohol by heat. —A. Quet.

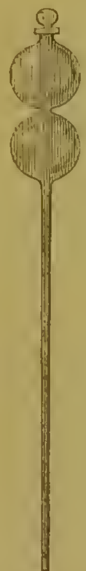
Perrot states that when the electric sparks are passed for a long time through alcohol vapour, the alcohol is at length completely decomposed into a gas (containing no ethylene, but which is absorbed by bromine) and a solid residue consisting of charcoal, with a trace of resinous matter. The bromine solution of the gas is colourless, has a sweet taste, and an odour somewhat similar to that of chloroform.

Alcohol has never been solidified. Faraday exposed alcohol to a temperature of 160° Fahr. below zero; it thickened, but did not congeal. Hence the great use of spirit thermometers when a very low temperature is required to be noted.

Faraday obtained this exceedingly low temperature by the following means. He prepared a bath of Thilorier's mixture of solid carbonic acid and ether in an earthenware dish, of the capacity of about 4 cubic inches, which was fitted into a similar dish somewhat larger, with three or four folds of dry flannel intervening. Such a bath will retain the carbonic acid in a solid state for about twenty minutes. To procure a still lower degree of cold, the bath of carbonic acid and ether was put into an air pump, and the air and gaseous carbonic acid rapidly removed. Operating in this way, the temperature fell so low that the vapour of carbonic acid given off by the bath, instead of having a pressure of one atmosphere, had only a pressure of $\frac{1}{24}$th of an atmosphere, or 1·2 inches of mercury, for the air pump barometer could be kept at 28·2 inches when the ordinary barometer was 29·4. At this low temperature the carbonic acid mixed with the ether was not more volatile than water at the temperature of 86° Fahr. (30° C.), or alcohol at ordinary temperatures.

Alcohol has a powerful affinity for water; hence the necessity of keeping it in ground stoppered bottles. When mixed with water, much heat is produced, and a diminution in volume takes place. This may be shown by mixing the two liquids in the apparatus shown at Fig. 2—which consists of a long tube of glass, furnished with two bulbs on the upper part, and having the elongated neck of the higher bulb closed perfectly with a ground glass stopper. The tube and lower bulb are filled with distilled water; when this is effected, alcohol is poured in till the upper bulb is filled; the stopper is next replaced, and the tube gently inverted. The two liquids now combine, and in the tube an empty space is visible, which before combination was completely filled. The space unoccupied shows the amount of contraction in bulk which has arisen from the combination of the alcohol and water; an elevation of temperature also takes place, in consequence of the chemical combination and the diminished specific gravity of the mixture. Thus, equal measures of alcohol, of spec. grav. 0·825, and water, each at 50° Fahr. (10° C.), when suddenly mixed, rise to the temperature of 70° (21° C.); and a mixture of equal parts of proof spirit and water, at 50° Fahr. (10° C.), give, under the like circumstances, a mixture having a temperature of 60° Fahr. (15°·5 C.) When alcohol and ice are mixed, the temperature is considerably reduced. Absolute alcohol, with a little more ice than it will dissolve, reduces the temperature as low as —35° Fahr. (—37°·2 C.). Spirit of wine, of spec. grav. 0·86, and 61° Fahr. (16° C.) temperature, mixed with snow at 32° Fahr. (0° C.), is cooled down to —14° Fahr. (—25°·5 C.). The contraction arising from the admixture of alcohol and water, increases regularly till the liquid consists of nearly one atom of alcohol and three atoms of water ($C_2H_6O + 3H_2O$), or 52·3 volumes of alcohol and 47·7 volumes of water, which in mixing contract to 96·35 volumes; hence the contraction is 3·65 volumes.

Fig. 2.



Thillaye shows that the mixture, when water is present beyond a certain limit, expands sensibly; his experiments also prove, besides, that when three volumes of alcohol, of spec. grav. 0·964, are mixed with seven of water, the mixed solution has a spec. grav. of 0·9850, whereas the calculated specific gravity should be 0·9863, thus indicating a decrease of gravity and a corresponding increase of volume, amounting to ·0013. This expansion, however, is only apparent, on account of the heat which is generated; but if the mixture were made of absolute alcohol and water till the specific gravity became 0·985, instead of an expansion, a contraction of 0·007 would be observed.

The following table, from the calculation of Gay-Lussac's experiments by Rudberg, shows the contraction of every decreasing 5 per cent. in the content of alcohol:—

Per cent., in volume, of absolute alcohol in 100 volumes of mixture at 59°.	Contraction, in per cent., of the volume of the mixture.	Per cent., in volume, of absolute alcohol in 100 volumes of mixture at 59°.	Contraction, in per cent., of the volume of the mixture.
100	0·00	50	3·74
95	1·18	45	3·64
90	1·94	40	3·44
85	2·47	35	3·14
80	2·87	30	2·72
75	3·19	25	2·24
70	3·44	20	1·72
65	3·61	15	1·20
60	3·73	10	0·72
55	3·77	5	0·31

From this table it will be observed, that the contraction is the same with mixtures containing different amounts of alcohol; for example, with the mixture containing 70 per cent. of alcohol and that containing 40 per cent. the contraction is 3·44. The reason is evident: the contraction increases to a certain point, and then decreases as the proportion of absolute alcohol lessens, giving to the intermediate mixtures, between the maximum and minimum points, a corresponding degree.

The volatility of alcohol is generally affected by admixture with water, as well as its specific gravity and expansive force. Tralles found that small quantities of water mixed with alcohol do not sensibly raise the boiling point of the liquor beyond that of pure alcohol; and Soemmering has shown that a mixture of alcohol with about 3 per cent. of water has greater volatility than absolute alcohol, and that a spirituous liquor compounded of 94 per cent. of absolute alcohol and 6 of water, possesses the same volatility as alcohol of 0·7947. Further, according to Soemmering, when alcohol of 0·7947 density is mixed with water till the specific gravity becomes 0·8, and distilled, those portions which first pass off are saturated with water, and the alcoholic solution in the retort becomes richer, till, in the end, absolute alcohol passes over; on the contrary, when the mixture contains over 6 per cent of water, the first portions of the distillate are richest in alcohol, and afterwards they become weaker to the end of the operation. The temperature also rises as the alcohol is expelled, gradually approaching to that of boiling water, and actually attaining that point at the close of the process, when all the spirit is driven over.

Taking advantage of this property, an attempt has been made to give the strength of various mixtures of alcohol and water, by ascertaining the temperature of the vapour, for which purpose Groening has constructed the following table. It consists of three columns: the first shows the temperature; the second, the quantity of alcohol in the boiling solution; and the third, the quantity of alcohol in the vapour evolved:—

Temp. Fahr.	Per cent. of alcohol in the boiling liquid in the retort.	Per cent of alcohol in the distillate.	Temp. Fahr.	Per cent. of alcohol in the boiling liquid in the retort.	Per cent. of alcohol in the distillate.
171°	92	93	189°·5	20	71
171·4	90	92	191·8	18	68
172	85	91·5	194	15	66
172·7	80	90·5	196·3	12	61
173·8	75	90	198·5	10	55
175·1	70	89	200·8	7	50
176	65	87	203	5	42
178·3	50	85	205·3	3	36
180·5	40	82	207 5	2	28
182·8	35	80	209·8	1	13
185	30	78	212	0	0
187·3	25	76			

The elastic force of alcohol vapour is very great; the following table shows the elastic force from 32° to 264° Fahr. (0°·0 to 128°·8 C.). The specific gravity of the alcohol was 0·813.

Temperature. Cent.	Temperature. Fahr.	Force of vapour.	Temperature. Cent.	Temperature. Fahr.	Force of vapour.	Temperature. Cent.	Temperature. Fahr.	Force of vapour.
0°·0	32°	0·40	57°·2	135°	12·15	102°·2	216°	72·20
4°·4	40	0·56	60°·0	140	13·90	104°·4	220	78·50
7°·2	45	0·70	62°·7	145	15·95	107°·2	225	87·50
10°·0	50	0·86	65°·5	150	18·00	110°·0	230	94·10
12°·7	55	1·00	68°·3	155	20·30	111°·1	232	97·10
15°·5	60	1·23	71°·1	160	22·60	113°·3	236	103·60
18°·3	65	1·49	73°·8	165	25·40	114°·4	238	109·90
21°·1	70	1·76	76°·6	170	28·30	115°·5	240	111·24
23°·8	75	2·10	78°·3	173	30·00	117°·7	244	118·20
26°·6	80	2·45	81°·1	178	33·50	118°·4	247	122·10
29°·4	85	2·93	82°·2	180	34·73	120°·0	248	126·10
32°·2	90	3·40	83°·3	182	36·40	121°·1	250	132·30
35°·0	95	3·90	85°·0	185	39·90	122°·2	252	138·60
37°·7	100	4·50	87°·7	190	43·20	123°·3	254	143·70
40°·5	105	5·20	89°·4	193	46·60	125°·5	258	151·60
43°·3	110	6·00	91°·1	196	50·10	126°·6	260	155·20
46°·1	115	7·10	93°·3	200	53·00	127°·7	262	162·40
48°·8	120	8·10	96°·6	206	60·10	128°·8	264	166·10
51°·6	125	9 25	98°·8	210	65·60			
51°·4	130	10·60	101°·1	214	69·30			

The expansion of alcohol by heat is not uniform; 1000 measures, spec. grav. 0·817, become 1079 when heated from 50° to 170° Fahr. (10° to 76°·6 C.). At a medium temperature the expansion is a little below the true mean; but with the state of dilution of the alcohol this difference between both ends of the scale becomes more marked. The greatest uniformity of expansion is between —14° and +98° Fahr. (—25°·5 and 36°·6 C.), being about 0·00047 of its volume for every degree. The contraction of alcohol from its boiling point, 173°·1 Fahr., has been investigated by GAY-LUSSAC, who gives the condensation of 1000 volumes in every 9° Fahr., or 5° C., from the boiling point of the liquid.

Temperature. Centigrade.	Temperature. Fahrenheit.	Volume of alcohol.	Temperature. Centigrade.	Temperature. Fahrenheit.	Volume of alcohol.
78°·4	173°·1	1000·0	38°·3	101°	951·3
73°·3	164	994·1	33°·3	92	949·1
68°·3	155	988·6	28°·3	83	944·0
63°·3	146	982·5	23°·3	74	939·0
58°·3	137	975 7	18°·3	65	934·0
53°·3	128	970·8	13°·3	56	929·3
48°·3	119	965·3	8°·3	47	921·5
43°·3	110	969·7	3°·3	38	919·9

One volume of alcohol yields 488·3 volumes of vapour at 212° Fahr. (100° C.); compared with water at the same temperature, the volume of alcoholic vapour is greater in the ratio of 3·11 to 1·00.

Alcohol vapour, when transmitted through a red-hot porcelain tube, is decomposed. SAUSSURE found that, on passing it slowly through a porcelain tube heated to redness, a little carbon was deposited on the interior of the tube, together with a volatile crystalline substance—naphthaline—and a brown empyreumatic oil; the gaseous products were carbonic oxide, carbonic acid, hydrogen, marsh gas, olefiant gas, and aqueous vapour. M. BERTHELOT passed alcohol vapour through a porcelain tube filled with pumice, and heated to redness. He obtained carbon, hydrogen, marsh gas, olefiant gas, aldehyde, naphthaline, benzol, phenyl hydrate, and (?) acetic acid.

The analysis of alcohol by SAUSSURE, DUMAS, and others, shows that it consists of—

	Atomic weight.	Per cent.
2 Eqs. of carbon,	24	52·18
6 Eqs. of hydrogen,	6	13·04
1 Eq. of oxygen,	16	34·78
	46	100·00

Formula:—C_2H_6O.

Alcohol possesses the property of absorbing gases in even a higher degree than water. Many experiments have been made by SAUSSURE on this subject, from whose results the following table has been formed:—

Absorbed at 64°·4 Fahr. (18° C.).	By 1 volume air-free water.	By 1 volume air-free alcohol. sp. gr. 0·84.
	Volumes.	Volumes.
Sulphurous acid gas,	43·78	115·77
Sulphuretted hydrogen,	2·53	6·06
Carbonic acid,	1·06	1·86
Nitrous oxide,	0·76	1·53
Olefiant gas,	0·155	1·27
Oxygen,	0·065	0·1625
Carbonic oxide,	0·062	0·145
Hydrogen,	0·046	0·051
Nitrogen,	0·042	0·042

Alcohol absorbs about 68 volumes of hydrochloric acid gas; it also takes up considerable quantities of cyanogen and nitric oxide. Most of these gases are evolved when the saturated spirit is boiled or exposed to the air, while others, such as hydrochloric acid gas, nitrous acid, &c., decompose the liquid.

Strong alcohol is one of the best solvents which the chemist possesses; by dilution with water this property is much diminished. Sulphur is dissolved by it when hot; but is deposited again in small crystals as the solution cools. An alcoholic solution of sulphur becomes turbid when diluted with water, and evolves a peculiar hepatic odour, and like the soluble sulphides precipitates metals as sulphides. Phosphorus is also dissolved by alcohol, for which purpose BUCHNER states that 320 parts of cold and 240 part of hot alcohol are required. From the latter solution one-fourth of the quantity of phosphorus deposits as it cools.

Alcohol absorbs chlorine gas with great avidity; it loses part of its hydrogen, which is replaced by the chlorine, forming hydrochloric acid, aldehyde, acetic acid, ethyl acetate, ethyl chloride, and chloral. When the action is long continued this last substance is the principal product

$$\overset{\text{Alcohol.}}{C_2H_6O} + 4Cl_2 = \overset{\text{Hydrochloric acid.}}{5HCl} + \overset{\text{Chloral.}}{C_2HCl_3O}.$$

With bromine it acts in a similar manner, forming bromal and hydrobromic acid. The heat evolved in these reactions often causes the alcohol to take fire.

Iodine is dissolved by alcohol, and forms a brown coloured solution; if hot alcohol be employed, the iodine is deposited in crystals after some time, and hydriodic acid is formed in the liquid. If to a solution of iodine in alcohol, an alcoholic solution of potassium hydrate be added, the fluid becomes colourless, and iodide of potassium and iodoform are simultaneously produced,

By a corresponding decomposition, when distilled with hypochlorite of lime, or bleaching powder, alcohol gives rise to choloform.

$$\overset{\text{Ethyl alcohol.}}{2C_2H_6O} + \overset{\text{Calcium hypochlorite.}}{5CaCl_2O_2} = \overset{\text{Chloroform.}}{2CHCl_3} +$$
$$2CaCO_3 + 2CaCl_2 + CaH_2O_2 + 4H_2O.$$

Concentrated chloric, bromic, and nitric acids react violently upon alcohol, producing acetic acid and a number of other bodies. This reaction is sometimes so energetic as to cause combustion. A mixture of very weak sulphuric acid and peroxide of manganese, when distilled with spirit of wine, yields principally aldehyde; acetic and formic ether are also formed, and towards the end of the distillation a weak solution containing acetic and formic acids passes over.

Pure chromic acid acts upon alcohol, giving rise to aldehyde and chromium sesquiacetate. Sulphuric acid, with moderately dilute alcohol, gives rise to ether, as also do phosphoric and arsenic acids; even selenious acid, on being distilled with that liquid, causes the formation of ether. On distilling a mixture of selenious acid, sulphuric acid, and alcohol, the distillate emits a most horrible odour; evolving hydro-selenic acid (selenetted hydrogen, H_2Se), and selenium deposits in the retort. Acetic, oxalic, formic, hydrochloric, hydrobromic, and hydriodic acids decompose alcohol, giving rise to ethers. Chloride of antimony, sesquichloride of iron, bichloride of tin, &c., decompose alcohol, forming oxides and hydrochloric ethers. Potassium behaves with alcohol as with water.

GRAHAM has shown that alcohol combines with most salts, forming with them peculiar compounds, alcoholates, analogous to hydrates. A great many of the neutral salts are soluble in alcohol; in general, all deliquescent salts, excepting the carbonate of potassa, are soluble in alcohol, and those inorganic compounds which are only sparingly soluble in water also prove insoluble in that liquid.

Alcohol is peculiarly adapted for dissolving copal, mastic, and a great number of resins which are employed in the preparation of the finest varnishes. The fats and volatile essential oils are likewise dissolved by it, and are employed with balsams in the composition of an extensive class of tinctures.

Anhydrous alcohol completely stops the current of a weak voltaic battery. CONNEL states that if two parallel platinum plates be introduced as electrodes into alcohol of spec. grav. 0·790, at 68° Fahr. (20° C.), their surfaces being $\frac{1}{10}$th of an inch apart, and the current of a battery of 216 pairs of 4-inch plates made to act on the liquid, no gas is evolved, but a little resin deposited at the positive pole, whilst pure hydrogen escapes very slowly at the negative pole. LÜDERSDORFF found that alcohol of spec. grav. 0·789 acted in a similar way under the influence of a powerful electric current.

The more dilute the alcohol, the less resistance does it offer to the passage of the current, and the greater is the quantity of hydrogen evolved at the negative pole; when the quantity of water present is comparatively large, oxygen is likewise evolved at the positive pole. Aldehyde, acetic acid, and other products of decomposition are produced at the same time.

The imperfect combustion of alcohol by means of platinum has already been noticed (see ACETIC ACID). When platinum black is shaken on paper moistened with strong alcohol, it makes a hissing noise, and becomes red hot, and if it does not set fire to the alcohol, continues to glow, and produce acetic acid as long as there is any alcohol.—EDMUND DAVY. If the platinum black be first moistened with water or alcohol, the ignition of the alcohol is prevented, and it is entirely converted with evolution of heat into acetic acid.—DÖBEREINER.

Sir HUMPHREY DAVY discovered that a fine platinum wire, heated to redness, and held in the vapour of ether, continued ignited for some time. Mr. GILL has practically applied this discovery in the formation of an alcohol lamp—lamp without flame—of the following construction:—A cylindrical coil of thin platinum wire is placed, part round the cotton wick of a spirit lamp, and part above the wick, and the lamp lighted to heat the wire to redness; on the flame being extinguished the alcoholic vapour keeps the wire red-hot for any length of time, according to the supply of the spirit, and with a very small expenditure thereof, so as to be in constant readiness to ignite tinder or a lucifer match. The proper size of the platinum wire is the hundredth part of an inch, which may be easily ascertained by coiling ten turns of the wire on a cylinder, and if they measure the tenth of an inch it will be right. A larger size only yields a dull red light, and a smaller one is difficult to use; about twelve turns of the wire will be sufficient, wrapped round any cylindrical body suited to the size of the wick of the lamp, and four or five coils should be placed on the wick, and the remainder of the wire above it.

A lamp constituted as above will require about half an ounce of alcohol to keep it in readiness for eight hours. This lamp affords sufficient luminousness to show the hour of the night by a watch, and to perform other useful services.

Alcohol, under the influence of concentrated sulphuric acid, may be converted, with the aid of heat, into water and olefiant gas ($H_2O_4 + C_2H_4 = C_2H_6O$). It was discovered by HENNEL, and confirmed by BERTHELOT, that it is possible to reunite these bodies.

FARADAY observed that sulphuric acid absorbed as much as 40 times its volume of olefiant gas. The resulting compound is sulphovinic acid, or as it is now termed, ethyl sulphuric acid ($C_2H_6SO_4 = H_2SO_4$, in which half the H is replaced by ethyl, C_2H_5). HENNEL added water to this body, and on distilling the mixture obtained ethyl alcohol.

BERTHELOT dissolved 30 litres of pure olefiant gas in 900 grammes of pure and very concentrated sulphuric acid. He afterwards added to the sulphuric acid 5 or 6 volumes of water, filtered and distilled it; then again submitting the liquid to several successive distillations, and putting it, after each distillation, in contact with potassium carbonate, in order to separate the aqueous portion of the distillate, he obtained 52 grammes of an alcohol corresponding to 45 grammes of absolute alcohol, and representing three-fourths of the olefiant gas; the remainder was lost in the manipulations.

The alcohol thus formed possessed all the properties of ordinary alcohol—the same taste and spirituous odour, the same boiling point, the same inflammability and the same colour in the flame, the same solvent power, the same reaction on all bodies and formations of absolutely identical products.

SAYTZEFF endeavoured to convert acetic acid into ethyl alcohol by treating it with deoxidizing substances, but without success. He, however, turned his attention to acetic chloride, or acetyl chloride (C_2H_3OCl), as the only derivative that can be readily prepared from it in large quantity, and that more easily undergoes chemical changes than the acid itself.

For the conversion of acetic chloride into ethyl alcohol a substance had to be chosen which, in presence of sodium amalgam, evolved hydrogen without decomposing the acetic chloride. As such a body he employed glacial acetic acid, and by acting on a mixture of this substance and acetic chloride with solid sodium amalgam, he obtained ethyl alcohol in considerable quantity.

The conversion of this mixture of acetic chloride and acetic hydrate proceeds in this way:—The hydrogen generated by the action of the sodium amalgam on the acetic hydrate apparently changes the acetic chloride into aldehyde, which by the further action of hydrogen passes into alcohol; a fresh quantity of acetic chloride then converts the alcohol into acetic ether, which is the final product of the reaction. Along with this process another reaction occurs, whereby, at the expense of the acetic chloride and sodium acetate, acetic anhydride is formed, and this by the nascent hydrogen is also converted into alcohol, which, as in the first case, is obtained as acetic ether.

The operation is conducted in the following way:—Solid sodium amalgam, finely divided, and consisting of 100 parts of mercury and 3 parts of sodium, is placed in a flask fitted to the lower end of a condenser and immersed in ice water. To $1\frac{1}{2}$ molecules of the sodium a mixture of 1 molecule of acetic chloride and 2 molecules of acetic hydrate is taken, and is gradually poured upon the amalgam, the contents of the flask being constantly agitated either by shaking or by a glass stirrer passed through the cork. After running in all the mixture the flask is kept in motion until its contents become a solid mass, when it is set aside for twenty-four hours. Water is then added and the mixture distilled until oily drops cease to come over with the water. The distillate consists of two layers, the upper of which has a strong odour of acetic ether. This is decomposed by concentrated potash solution; and an alcoholic fluid is then distilled off, which after rectification and drying is anhydrous ethyl alcohol.

LINNEMANN has obtained ethyl alcohol by acting on acetic anhydride with sodium amalgam. The acetic anhydride is added to an amalgam containing not more than 4 per cent. of sodium, with very great caution, avoiding both a rise of temperature and the hardening of the mass, which at the end of the operation should appear dry and dusty. It is then mixed with powdered ice or snow, when the amalgam deliquesces without evolution of gas. Water and a little more amalgam are then added, an oily matter is separated by filtration, and the mixture neutralized with solid potassium carbonate. The liquid is then rectified in the usual way, when pure ethyl alcohol results.

In those cases where it is desired to determine with certainty the existence of alcohol in presence of a large quantity of water, BERTHELOT recommends the following method. Benzoyl chloride (C_7H_5OCl) is decomposed very slowly by cold or lukewarm water; but if the water contains alcohol a benzoic ether is immediately formed; the ether is found with the excess of benzoyl chloride. The presence of the ether can be made manifest by heating a drop of the benzoyl chloride with aqueous solution of potash, which dissolves the acid chloride almost immediately, without acting at first on the ether. The reaction is very distinct with $\frac{1}{100}$th part of alcohol. Even with a $\frac{1}{1000}$th part of alcohol, the smell of the benzoic ether is very apparent when a few cubic centimètres of liquid are used.

ALCOHOLIC LIQUORS—*Whisky; Usquebaugh; wysge*, a stream; *uisge-béatha*, water of life, Irish; *usqueba*, French; the spirit distilled from barley, rye, wheat, &c. The Latin epithet, *aqua vitæ*, the Irish term usquebaugh, and the modern word whisky, are in point of fact synonymous. The English, shortly after the invasion, in the time of HENRY II., found the Irish people indulging in potations of this liquor. History informs us that the knowledge of *aqua vitæ* was first acquired in Europe in the reign of that monarch; but it is more than probable that the Irish were acquainted with it before the English. The strong similarity between the Irish language and the primitive languages of Asia, as stated by eminent etymologists, and the intercourse the Irish had with that quarter of the world, lead to the supposition that the art of distillation was introduced directly from India.

CAMPION relates, that when the new settlers were attacked by any of the diseases common to the country, they used *aqua vitæ*, or *usquebaugh*, the ordinary beverage of the inhabitants, as the best restorative of health.

The directions for making *aqua vitæ* or *usquebaugh*, and liquors compounded from it, are recorded in the Red Book of Ossory, a work compiled about 500 years since. *Aqua vitæ* was first issued as a medicine, and was considered as a panacea for all disorders; the physicians recommended it to patients indiscriminately, for preserving health, dissipating humours, strengthening the heart, curing colic, dropsy, palsy, &c., and even for prolonging existence itself beyond the common limits. Hence it was eagerly sought, and the taste, once formed, has been transmitted from generation to generation.

The excise duties have great influence on the mode of manufacture of spirits in this country. The system of supervision is a remarkable instance of excise machinery—a supervision rendered important by the great sum collected annually, considering the comparatively small number of distillers by whom the payments are made. As the duty per gallon is estimated on spirit of one particular degree of strength, the greatest caution is necessary in testing all the liquors produced, as a guarantee that every sample is charged exactly in proportion to its quantity and to its strength. Excise officers, as agents for the government, are almost constantly present at every distillation, day and night. They succeed each other, one or more at a time, as may be necessary, after intervals of eight hours; the periods being from six A.M. to two P.M.; from two P.M. to ten P.M.; and from ten P.M. to six A.M. of the following day. The Act of Parliament to regulate distilleries was passed in 1825, and by its provisions no distiller was permitted to work till he had procured a license, which was to be renewed yearly; moreover, he was not allowed to have on his premises a still below a certain capacity. The number of stills, charges, receivers, &c., continues subject to certain restrictions; and the exact routine is given as to the mode the liquid shall run from one vessel to another in the process of distillation. The openings in the principal vessels are expressly stated, and the most scrupulous care is taken that nothing shall pass from one vessel to another without traversing a pipe having a lock or valve, which is provided and kept in repair by the distiller, to the satisfaction of the excise officer, who has charge of the key, under a penalty of £200. This functionary also keeps the keys to lock up the furnace doors and stills; in fact, he exercises a perfect control over every operation and process; and with the view of facilitating his superintendence, the brewing and the distilling take place at different periods, one portion of time being set apart for the preparation of the *wash*, and another for its distillation.

In order that the intentions of the law may be fully carried out, prohibiting the synchronous brewing and distilling, the buildings are detached or conveniently divided, and the pipes, a large number of which are visible, are of various colours. The legislature requires that the conduit pipes shall be painted *black;* those for the conveyance of wash, *red;* those for the first distillate, *blue;* and those for the finished spirit, *white;* this is done in order that the officer may conveniently trace the routine of the processes; further, ladders and all other conveniences must be furnished for the easy access of the supervisor to all the vessels in the establishment.

The new Act specifies that no spirit receiver shall be used in any distillery, which shall not be made, placed, and fixed, to the satisfaction of the commissioners of Inland Revenue, and be sufficiently deep to admit of the gauge being taken of the depth of 15 inches in the centre; and every such receiver shall be so filled, that at the time of gauging the same, for the purpose of charging the duty thereon, the depth of the spirit shall not be less than 15 inches, under a penalty of £50.

The fabrication of ardent spirits is a very extensive branch of the home trade of this country. Whisky is the staple produce; gin, rum, brandy, &c., being made in other countries by operations analogous to those followed in the manufacture of whisky. Gin and rum are extensively manufactured in this country, but invariably from whisky.

The operations of a *distillery* relate to the extraction of the alcohol from various sorts of grain. Wheat, oats, barley, rye, Indian corn, rice, and other grains, whether in the raw or in the malted state, as well as the juices of fruits, sugar-cane, beetroot, potatoes, carrots, and even some of the *grasses*, and many other vegetable and natural substances, by peculiar processes are made to yield alcohol. Distillation is invariably one of these operations, but it is preceded by others which differ according to the nature of the ingredients employed.

Those universally known liquors, whisky, hollands, gin, brandy, rum, spirits of wine, and cordials of various kinds, all contain alcohol, which passes over in the process of distillation. British brandy, British gin, whisky, or rum, are produced from corn; French brandy, from wine; West Indian rum, from sugar or molasses. The different qualities of these various liquids depend partly on the percentage of alcohol contained, partly on the berries, herbs, and seeds with which they are flavoured, partly on their mode of manufacture, and lastly, on the substances whence they are derived. In every case, however, the substance distilled is a *sweet* liquid, though the means by which the saccharine material is formed vary with circumstances. The extract produced from grain is fermented before being distilled.

This liquor, modified in a particular way at the brewery, constitutes *beer;* in the distillery it is known under the name of *wash*, and is the liquid which undergoes, subsequently, the process of distillation.

The first objects that meet the eye in a large distillery are the magazines in which the grain is stored; beyond these are the various buildings connected with the still-room. Near the entrance are the mill and the brewhouse.

All the grain goes through the granary, and when required in meal, passes into the mill-room, which contains several pairs of millstones, worked by a large steam-engine. The meal next goes to the brewhouse, in which are large coppers for heating water, and prodigious mash-tuns, capable of holding many thousand gallons. Here it is mashed, its residue being sold to the cattle-feeders. The coolers adjoin the brewhouse.

From the coolers the wort descends into what are termed the *fermenting backs*, a series of square vessels of enormous proportions, where it is exposed to the action of the yeast. The alcoholic fermentation here commences with great energy, and continues till the sugar of the sweet wort has been transformed into alcohol. By subsequent distillation from large copper *wash* stills, this alcohol is obtained in a more concentrated state, forming the *singlings* or *low wines* of the distiller.

For the production of whisky, barley is the most abundantly employed of all the cereals, either in the raw or malted state. Malting is a preliminary operation to which the barley is submitted by those who employ malt; but since it is not *solely* used by the distiller, the detailed description of the operations of malting will be considered under the article BEER. To show, however, the influence the malting has on the operations of the distiller, it is necessary to notice one particular change that takes place in the constituents of the grain—namely, the metamorphosis by which first diastase, and afterwards sugar, is produced. Diastase is a peculiar nitrogenous substance, formed during the germination of grain, potatoes, &c. Its proportion in malt does not exceed 0·002 to 0·003 per cent.; but nevertheless it is solely to the presence of this body that malt owes its value as an agent for determining the conversion of starch into grape sugar.

The change of starch into sugar is partly effected in the grain during the time it is permitted to sprout, but principally after it has been infused in water, when the diastase, which has been formed during germination, acts as a ferment on the starch of the grain, and converts the whole of it into glucose (grape sugar, $C_6H_{12}O_6$), which, when fermented, splits up into ethyl alcohol and carbonic acid—

$$\overset{\text{Grape sugar.}}{C_6H_{12}O_6} = 2CO_2 + \overset{\text{Ethyl alcohol.}}{2C_2H_6O}.$$

This conversion of the nutritive parts of the grain disintegrates it in such a manner that water readily permeates its entire substance, and takes up the whole of the soluble matter. For this reason malted grain is preferred by many distillers: firstly, because the extract is obtained more perfectly and with greater facility; and secondly, because it is supposed that the yield of spirit is larger than would be produced were the grain unmalted. But as additional labour always involves greater expense, the cost of the malted substance is necessarily higher, besides that the government duty on malt further raises its value. To avoid this expenditure many distillers use a mixture of malted with raw or unmalted grain in various proportions; and though the subsequent extraction and management of the worts from such mixtures present some difficulties, yet they are for the most part overcome by care and foresight, and the yield of spirit is as large as if malt only was taken.

Whether malt is exclusively used or a mixture, the substance must be either ground or crushed, in order to expose an extensive surface to the solvent action of the water in the preparation of the extract. As said before, the malting effects this disintegration to a considerable extent; hence the reason why malt is not required to be in a very minute state of division when subjecting it to the action of solvents; but when mixtures are used, into the composition of which large quantities of raw grain enter, they must be finely ground for the cause just assigned.

Ground or crushed malt always yields a wort that is comparatively clear; mixtures, on the other hand, never give a bright wort, but dense solutions, on account of the large quantities of starch which they contain suspended mechanically in the water.

This behaviour of the malt and mixtures constitutes one of the principal differences between the brewer's and distiller's mode of making the worts; the former must have a clear extract, as the liquid, after being submitted to the slow fermentation, remains to give *body* to his beverage. Such an extract cannot be otherwise obtained than by using malt entirely. With the distiller, it is optional whether he uses malt or mixtures to prepare his worts, since he can run the starchy liquor into the fermenting tun, there to undergo conversion into sugar, and its subsequent alcoholic fermentation.

In other respects the operations of the brewer and of the distiller are closely allied, excepting that the distiller's object is to urge fermentation to its utmost, and finally to separate by distillation the spirit thus formed from the wash, after which the residuary liquor is accounted of little value; whilst the brewer's aim is to prevent the fermentation going beyond a certain point, the alcohol produced being left in conjunction with the wash, and forming beer.

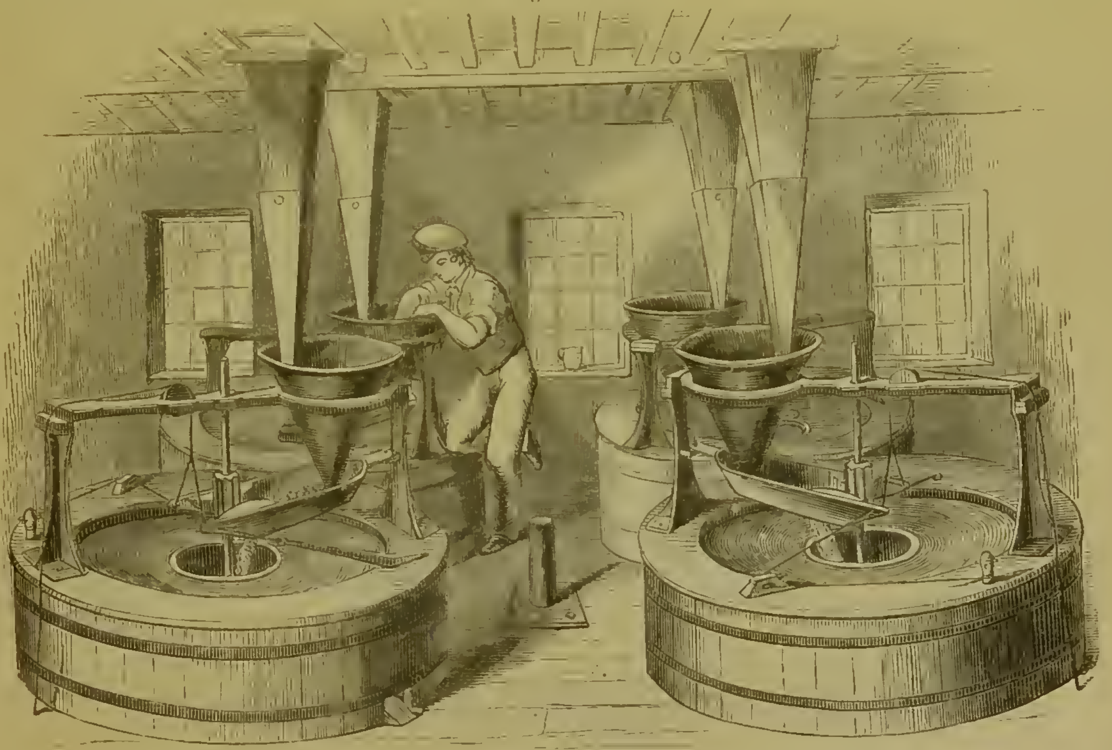
Considerable attention is required of both parties, and especially of the distiller who employs raw grain, in preparing the worts, owing to the tendency of the mixture *to set*, which prevents him from extracting the valuable portions. The main point, however, in the distiller's business, demanding particular care, is the proper management of the fermentative action succeeding the making of the worts, to insure the conversion of the whole of the saccharine matter into alcohol; the acetous fermentation must also be guarded against, and various others difficulties, which, if overlooked, would be extremely detrimental.

The several stages in the manufacture will now be fully treated of under the heads—GRINDING, MASHING, FERMENTING, and DISTILLING.

GRINDING.—The granary is a large building of brick or stone, having three spacious stories on which the malt or raw grain is hoarded. One of the granary floors is appropriated to the kiln-dried barley, which lies spread in a stratum 5 feet thick, ready to be conveyed to the mill. When it is to be ground into meal, the grain is taken to a room immediately over the mill-chamber, and discharged through trap-doors into cloth sleeves, which conduct it to the hoppers leading into the mill-room. Fig. 3 shows the nature of the operations. These stones grind all the raw grain; while the malt is passed through a crushing-mill, consisting of two rollers placed nearly in contact. In the lower room is the mechanism by which the millstones are rotated in the room above, and also the pipes for conducting the meal from the grinders into sacks. The meal, as it issues from these pipes, is heated to about 100° Fahr. (37°·8 C.) by the mechanical friction of the stones.

MASHING.—The mash tun is made of cast-iron plates, firmly bolted together and circular in shape. Fig. 4 represents the mash tun at a large Scotch distillery: *a a* is the mash tun, 28 feet in diameter and 8 feet deep. It is furnished with a false bottom, pierced with holes like a strainer. From the middle of the tun rises a vertical shaft, *m*, set in motion by machinery, and thus revolving the mechanism, *d c c*. This apparatus, by rotating horizontally and vertically, effectually agitates the whole of the liquor in the tun: *d* is a rod extending from the pillar, *m*, to the edge of the tun, round which its outer extremity is carried on a toothed wheel, gearing into the teeth round the tun; it is driven by the bevel wheels, *n*. Connected with this rod, *d*, are two parallel rods, *c c*, which it carries round with it: to these rods are fixed bent cross bars, which also revolve in consequence of the double motion of the rods, *c c*, rotating on their axis at the same time that they turn round the shaft in the centre of the tun. In their mashing and churning action they much resemble the paddles of a steamer. The one rod is exactly over the other; the paddles attached to the lowest pass within 2 inches of the bottom. The platform, *k*, is so arranged that the men may have easy access to the tun; *f* is a door leading to the mill, where the grain is ground into grist; *b b*, two channels or sluices, for conducting the grist from the granary into the mash tun; *e e*, wash-backs, resting on rafters, &c., which project from the wall. The wash-backs are used for containing the weak worts drawn from the last two *sperges*, or mashings of the grist, after the principal extracts have been drawn off; they are pumped up into *e e*, and kept to macerate fresh grist in the next operation. Several large copper vessels, besides the tuns, are in use, for the purpose of heating the water for mashing the grist; they hold several thousand gallons, and are heated by a furnace.

Fig 3.

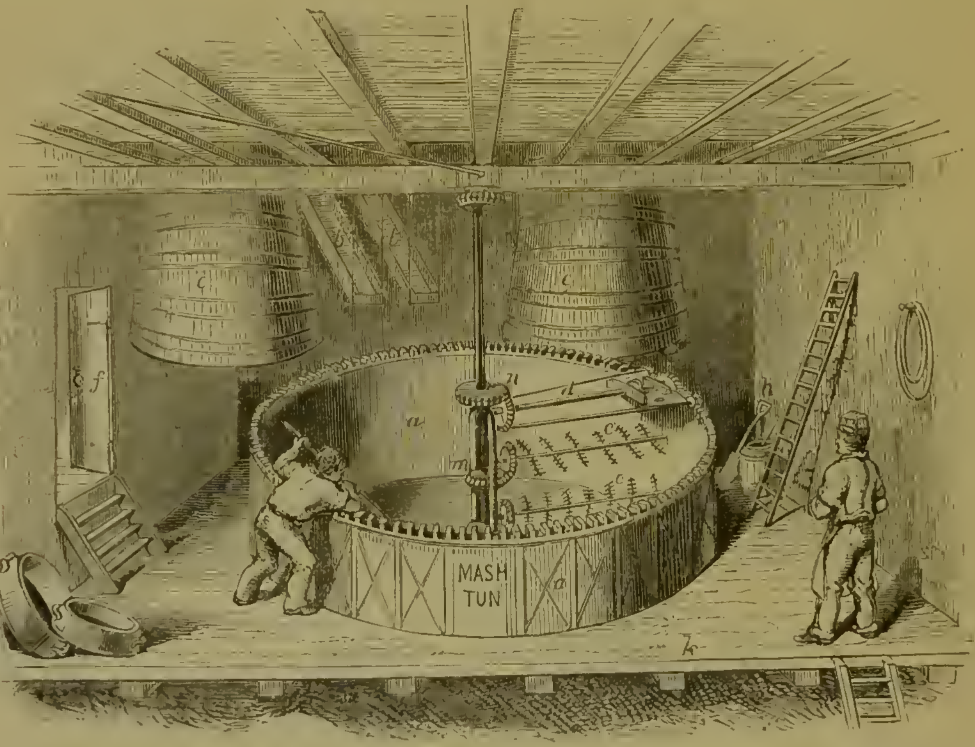


It has already been stated that the grist may be barley meal, oats, or malt, in variable proportions, according to circumstances. Large quantities of oats, on being mixed with the grist, confer a peculiar flavour, which is easily recognized by those who have experience in the taste of spirit. When barley malt and raw barley meal form the grist, 1 part of malt to 2 or 3 parts of the raw ground grain are considered the best proportions, though 1 part of malt to 5, 6, 8, 9, or even 15 parts of the raw grain are often used. When oats are employed the best proportion is 3 parts of barley malt to 1 of oats; but satisfactory results are obtained when malt and oats are used in the ratio of 2 of the former to 1 of the latter. If the proportion of raw grain be large when compared with that of the malt, it is a general custom to add a quantity of chaff, in order that, when the mashing water has extracted the saccharine and starchy portions, it may be more easily filtered or drawn off.

The quantity of malt and grain used at each mashing depends on the size of the distillery; hence no fixed rule can be laid down. In the distilleries in Dublin the quantity of grist at each brewing varies with circumstances, from 800 bushels, the lowest, to 2000 bushels, the largest quantity. In nearly all cases it is composed of seven-eight parts of raw or unmalted, and one-eight part of malted grain.

Previous to introducing the malt a quantity of water, at a temperature between 140° and 150° Fahr. (60° to 65°·5 C.), is run into the mash tun, and the ground malt and meal are then added. Workmen, with stout wooden spatulas or oars, keep the mixture in brisk agitation until the grist is thoroughly moistened, and neither clots nor lumps remain. In the larger establishments this part of the operation is effected by machinery. The perforated false bottom allows the wort to percolate into the space between it and the true bottom of the tun, from which it is drawn off more easily into the underbacks—large vessels placed beneath the mash tun, wherein the worts are collected till pumped into the cooling-backs. Distillers and brewers are more variable in their mode of working than any class of manufacturers who carry on business extensively. In no one operation do they seemingly follow a common rule, each having some favourite plan of his own of a supposed greater merit than others; hence the difficulty of giving a true and comprehensive detail of these branches. This assertion has been partly demonstrated already when speaking of the grist; and in the present operation—the mashing—it becomes still more manifest.

Fig. 4.



In France the mode of saccharification is to run in 6 to 7 hectolitres of water at the temperature of 50° C., then to add the 400 or 450 kilos. of malt, which gradually suck up the water. Lastly, by means of a pipe between the two bottoms, 36 hectolitres of water are run in at the temperature of 75° to 80° C.

During the operation of mashing the diastase dissolves in the tepid water and immediately reacts upon the starch of the malt (which has been modified and rendered more easy of conversion by germination), which it by degrees transforms into dextrine and glucose. An excess of diastase remains in the mixture, which is available for acting on raw grain, which may be now added if the temperature of the mash remains sufficiently high to determine the conversion of its amylaceous matters into starch.

In France and Belgium rice flour is added little by little at this part of the process, and subsequently beneath the false bottom a sufficient quantity of water, heated to 80° C., to raise the temperature of the whole mash to 70° C. Under these circumstances the rice starch is by degrees converted into sugar by the malt diastase. The whole is covered over and left for two or three hours, after which, to complete the saccharification, 40 hectolitres of water are run in beneath the false bottom. In an hour and half the operation is esteemed finished.

The brewer does not desire complete saccharification, some dextrine being necessary to him. The distiller, on the other hand, requires nothing but glucose in his mash.

The whole of the saccharine and fermentable matters of the grist introduced into the mash tun is generally extracted in three, always in four mashings at most; but the manner of doing so is different, according to the notions of the manager. In some cases the first, second, and third mashings are evaporated till the mixture acquires a spec. grav. of about 1·05, when it is thought to be ready for the fermenting tun, the fourth wash being reserved for extracting fresh quantities of grist. Other distillers employ such quantities of water in the first and second extracts as will allow the wort to be of a strength fit for fermenting; the third and fourth washings are then concentrated by evaporation to the proper density, and added to the first and second; or else are made of the proper strength by running them on fresh quantities of ground malt and grain. Others, again, manage the quantity of water in such a manner that the product of the first extract has the density necessary for fermentation, the remaining three washings being rendered stronger either by evaporation or mashing with fresh portions of malt or grist. In the latter case the quantity of water is larger in the first mashing.

In Macfarlane & Co.'s distillery at Glasgow 260 cwts. of grist, including a sixth or a fourth of malt, are taken for an ordinary mash. These are put into the mash tun, and about 788 barrels of water, nearly 28,368 gallons, are poured upon them at two stages of the operation. The first water is employed at 140° Fahr. (60° C.), the second water at 175° to 180° Fahr. (79° to 82° C.); the whole contents of the tun being brought to 150° Fahr (65°·5 C.).

In the Dublin distilleries about seven-eighths of raw grain are employed. The first mash is the only one let into the fermenting tun, the succeeding small worts being kept for the next day's brewing. In preparing the wort about 5 barrels of water are taken to the quarter of grist, but more if small worts are used; to completely exhaust the grist about the same amount of water is required for the last mashing.

The temperature of the water is made to depend on the quantity of malt present; when the malt and raw grain are mixed in the proportion of 1 of malt to 2 of grain, the first mashing may be made at a temperature of 150° to 160° Fahr. (65°·5 to 71° C.); but if the malt and grain be as 1 to 4, 6, or 9, then the water should not exceed 145° Fahr. (62°·5 C.) for the first mashing, in order to prevent the *setting* of the mass. The system pursued in France and Belgium, of adding the raw grain after the mashing of the malt is completed, obviates this difficulty. A longer time is likewise required for the first mashing with a large quantity of raw grain than if it were entirely malt. From one hour and a half to two hours generally suffice for this operation, when the contents of the mash tub are kept in agitation by machinery, and the proper heat of the water has been attended to; though sometimes the time occupied extends to three or more hours.

The time required for the wort to clarify, when it has been properly mashed, is about one to two hours. As the temperature of the solution becomes lower from contact with the grist and from the agitation, it is customary not to add the whole of the water employed in the first mash at once, but to retain from half to a quarter of the liquid, which is added at short intervals towards the middle of the operation. This serves to keep the heat more uniform, and the work is more effectually accomplished.

After the wort has been drawn off into the underback and pumped into the coolers, the second *sperge* is then let in upon the grist remaining in the mash tun, and a similar treatment to the foregoing given to the mash; the time occupied is one hour and a half, and the density of the weak wort is from 15 to 16 lbs. per barrel.

Since the greater part of the saccharine and starchy matters of the grist is extracted in the first infusion, there is but little danger of the mash setting in the second washing, so that the temperature of the water may be as high as 180° Fahr. (82° C.).

After this wort has been let into the under-back, the third and last sperge is made, and in about an hour and a half the wort is drawn off; its density is from 3 to 5 lbs. per barrel. Both these mashings are pumped into wash-backs, and are let down to form the first mashings in the next day's brewing, and thus the work succeeds by *three* sperges or waterings of the grist. In the case quoted, the first is drawn off at the proper density for fermentation, while the remaining two are retained to be further strengthened by additional extractive matter.

On tasting the solution during the infusion of the first wort, a very peculiar difference is observed. At first nothing particularly striking is apparent, but in a very short time the solution begins to acquire a sweetish taste, which becomes more and more perceptible, till the liquor possesses the lusciousness of malt worts. Hence it is evident that the starchy matter of the grist, as it is extracted, is resolved into glucose, or grape sugar. Two causes serve to effect this change: the first and chief agent is the peculiar body *diastase*, which is generated in the malt during the germination of the grain. This body possesses the property of converting much more starchy matter into sugar than is present in the malt. It reacts on the starch of the raw grain, and produces the same action as if the grain itself was malted. The gluten of the grain is capable of producing the same change, but it requires a considerably longer time than the active body in malt. When only 1 part of malted barley is used to 5 of the ground raw grain, the excess of diastase is sufficient to effect the transformation; but when 9 parts of the raw material are used to 1 of malt, the quantity of the active principle in the malt is inadequate to saccharify the whole of the starch, and a portion is consequently left till fresh diastase is formed. This accounts for the longer time and greater trouble involved in preparing the wort for the still when the proportion of raw grain is very large.

When a wort is being made, considerable time elapses before the starch is wholly transformed into sugar, and in the ordinary period allowed by the distillers in the fabrication of the worts, this conversion is never complete, a large portion of starchy matter remaining unaffected. Thus, if it be attempted to make a wort of 200 lbs. of saccharine matter per barrel in the ordinary way, the process fails, in consequence of the starchy matters assuming a jelly-like appearance long before the above strength is attained. In order to obtain the strongest possible wort from raw grain, the diluted extract should be left to repose till the whole of the starch has become soluble and saccharified, when the solution may be drawn off and concentrated by evaporation at a low temperature.

The specific gravity of the first wash is generally about 1·050; the second wash about 1·010 to 1·015; the third, 1·008; and the fourth a spec. grav. very little higher than that of water. The strongest wort procurable from grist, containing over 4 parts of unmalted grist, is obtained by evaporating the first mashing. The strength, even by this treatment, never exceeds 150 lbs. of saccharine matter per barrel, while the wort of malt, treated similarly, may be obtained of 200 lbs. to the barrel.

A wort of 62 lbs. per barrel, which would yield 12 per cent. of spirit of 1 to 10 strength, or 0·90917 spec. grav., is esteemed a good strength. The original Dutch hollands were obtained from a wort considerably weaker.

The whisky made by smugglers in Scotland was long preferred by the inhabitants, and was purchased at a higher price, under the name of Highland whisky. This was partly owing to its being made entirely from malt; but the chief reason was that, from the unfavourable circumstances under which they worked, their wort was necessarily much weaker than that of the legal distillers, and on an average was probably not much stronger than the wort of the Dutch hollands.

When malt alone is used, about 500 bushels, coarsely ground, are introduced in a mash tun of a proper size, and 9000 gallons of water, at a temperature of 160° Fahr. (71° C.), are run in upon it. The whole is kept in brisk agitation by machinery for an hour, then allowed to rest, and after the grains have subsided, about 6000 gallons of wort are drawn off into the coolers. From 4500 to 5000 gallons of water, at 180° Fahr. (82° C.), are then run in upon the residuary grains in the tun, and the mashing continued for about three quarters of an hour; the mixture is then allowed to rest, and the weak worts drawn off. A third treatment of the grain, with the same amount of water, takes up all the soluble matters of the grain; and when this has been drawn off, and the grains have been drained of all the solution, both the second and third mashings are mixed, constituting together 9000 gallons; this mixture is employed next day, at a temperature of 160° Fahr. (71° C.), to mash 500 bushels of fresh malt. When it has become saturated with saccharine matter, it is drawn off into the coolers as before shown, and the residuary grain subjected to the same operation as the grains of the preceding day.

Sometimes rye is used instead of malt: 90 bushels of it are mixed with 190 bushels of raw grain, thus constituting 280 bushels in the whole, for the mashing of which 5200 gallons of water are required. The temperature of this water, as is pointed out in the beginning of the remarks on this part of the subject, should not be so high as when all malt has been used; and as was also stated above, the time allowed for mashing is greater; the subsequent washings are reserved for exhausting fresh portions of grist.

The Cooling.—The worts have a great tendency to form acetic acid, which the distillers oppose by bringing the solution as speedily as possible to that temperature at which the alcoholic fermentation proceeds. Various methods are pursued by different distillers, according to the extent of the factory.

The coolers formerly used were shallow rectangular vessels, into which the first wort was pumped to the depth of 2, 3, 4, or more inches, as soon as it had passed the under-back. They were placed at the upper part of the building, one over the other, and were open on all sides to the winds. Over each cooler were fixed three *flighters*, machines like horizontal windmills, which, when made to revolve, swept a powerful air-current over the worts.

The time of cooling is now much shortened, and the waste of heat lessened, by causing the worts to pass through tubes surrounded by a stream of cold water. By this means, and the use of the proper extent of piping, the wort may be cooled down to the temperature of the surrounding water, or any other intermediate degree that may prove most advantageous. Worts cooled down by this means lose none of their water by evaporation, as they would do if cooled in the flat shallow wooden coolers; and consequently, with the exception of the little alteration in gravity, occasioned by the difference of temperature, the worts remain of the same density that they indicated when drawn from the underback.

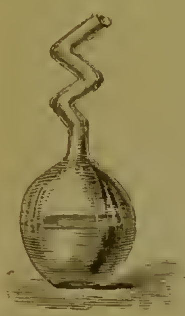
Fermentation.—When the clear juice of plants or fruits is exposed to the air for a few hours, at the temperature of 68° Fahr. (20° C.), it becomes turbid, and gives off carbonic acid; in other words, it begins to "work" or ferment, the temperature of the liquid at the same time becoming much higher than that of the air about it. This intestine change continues until the whole of the sugar is decomposed.

After the escape of gas has ceased, a multitude of small oval bodies, which do not exceed $\frac{1}{250}$th of an inch in diameter, and which are seen under the microscope to consist of nucleated cells, separates from the liquid. These cells are the ferment or yeast. Their presence is essentially necessary to vinous fermentation. *Bierhefe*, German; *ferment alcoolique*, French; *Torula Cerevisiæ*, botanic fungus.

It has been definitely established that the spores of yeast are universally diffused through the air, and that so soon as they meet with a solution containing the nutriment necessary for their development, yeast cells are produced, and fermentation sets in.

If air be excluded from grape juice or any saccharine solution, no fermentation takes place. Fresh grape juice may be kept for years at the temperature of 68° Fahr. (20° C.), without undergoing any change, provided it never comes in contact with the atmosphere: or if the air has been heated to redness, it may remain in contact with a saccharine liquor for an indefinite time without producing fermentation. But as Pasteur has shown, the motes floating in the air can be collected in cotton or asbestos placed in a tube through which air has been drawn: and a piece of this cotton or asbestos, placed in a sugar solution which has been well boiled, and cooled again, but which contains the mineral and albumenoidal constituents of yeast, develops fermentation. Sugar solutions containing the same yeast constituents, but without this air dust, undergo no alteration, neither do those in which cotton or asbestos alone is introduced. The same liquid remains unaltered if it has been boiled in a glass flask, the neck of which is so bent that dust cannot fall into it, the flask being afterwards left inclosed (Fig. 5.).

Fig. 5.



According to Helmholtz fermentation only takes place when the solution is sufficiently diluted with water. With less than 4 parts water to 1 part sugar it takes place but imperfectly, if at all; partly because the resulting alcohol precipitates the nitrogenous substances, destroys the fermentative power of the yeast, or renders the liquid unfit for its further development. If, on the other hand, the liquid is too dilute, the fermentation is slow, irregular, and readily passes into acetous fermentation. Moreover, it is essential that the yeast be in direct contact with the sugar solutions. A solution of sugar contained in a bladder, suspended within a fermenting liquid, does not ferment, but merely takes up alcohol by diffusion.

Payen states that an increase of yeast takes place in fermentation when the liquid, in addition to sugar, contains a nitrogenous substance. When, on the contrary, yeast is left in contact with a pure solution of sugar, it diminishes both in weight and fermenting power, and finally becomes totally inactive.

Pasteur mixed yeast with a saccharine solution and albumenoidal substances, and exposed the whole to the air in shallow vessels. He found that a quick and active increase of the yeast cells took place, but that only 6 to 8 parts of sugar were decomposed for each 1 part of the newly-formed yeast. When, however, a similar mixture was made and the air excluded, the yeast decomposed about 100 times its weight of sugar, but the yeast grew and multiplied very slowly.

Ménend fermented 100 parts of sugar with 20 parts of beer yeast, and obtained 13·7 pints of insoluble residue, which, when brought in contact with a larger quantity of sugar, diminished to 10 parts. The latter was white, resembled woody fibre, and was no longer capable of exciting fermentation. Hence it appears that the quantity of yeast must bear a definite proportion to that of the sugar; if the sugar is in excess, part of it remains undecomposed, or merely undergoes a very slow after-fermentation, often continuing for years. If, after all the sugar is decomposed, the fermented liquor still contains yeast, or nitrogenous matter not wholly converted into yeast, putrefaction will ensue.

Fermentation is the most important stage through which the materials in the hands of the distiller have to pass, and one which not only demands considerble skill and attention for its proper management, but also requires extensive knowledge, both of the principles of chemistry and of practical results. Indeed, the success of the operation almost entirely depends on the fermentation of the wort; and unless managed with due care and dexterity, a failure will be the consequence.

The proper fermentation of worts sometimes baffles the most skilful, the process being so changeable in its nature. Formerly the Scotch distillers used to add 3½ per cent. of fresh yeast, or 4 to 4½ and sometimes 5 per cent. by measure of stale yeast, to the wort in the fermenting tun. The whole of the yeast was not added at once, but at two, three, four, or more intervals. Usually 1¼ to 1½ per cent. of yeast was added the first day, and the remainder on the second and third days; though some distillers introduced the whole of the remaining yeast, after the first addition, on the third day, and, if requisite, a further quantity to complete the fermentation. Fresh yeast, if it could be obtained, was taken at first, and the succeeding quantity put in might be stale yeast; in which case a larger measure was employed than if the ferment were fresh.

The large amount of yeast formerly employed was in consequence of the great density of the worts. Distillers now generally use from 1 to 1½ per cent. of fresh yeast, very rarely 2 per cent. Of this about three-fourths per cent. are added when the wort is let into the tun, and the remainder after the second day—sometimes when the attenuation reaches 1·030 or 1·035. The quantity subsequently added may be stale yeast, taking, however, rather more than the usual allowance of the fresh ferment.

This yeast is invariably obtained from the large London porter breweries, the quantity produced in the provincial ones being insufficient. The yeast which is thrown off the porter during its fermentation is the best; though frequently the slimy dregs which remain in the bottom of casks when the clear porter is racked off is sold for yeast. A great deal of the success of the fermentation depends upon the goodness of the yeast, and the quantity used must be regulated by its freshness.

In the course of fermentation the liquid gains about 20 degrees Fahr. (4° C.) above its ordinary temperature. Donovan gives the following as a fair statement of the specific gravity and temperature of the wort of a five days' fermentation, the original specific gravity being 1·050:—

First morning,	temp.,	70°	Fahr.,	sp. gr.	of wort,	1·050
" evening,	"	70°	"	"	"	1·050
Second morning,	"	72°	"	"	"	1·046
" evening,	"	76°	"	"	"	1·032
Third morning,	"	80°	"	"	"	1·022
" evening,	"	84°	"	"	"	1·012
Fourth morning,	"	88°	"	"	"	1·007
" evening,	"	88°	"	"	"	1·005
Fifth morning,	"	88°	"	"	"	1·003
" evening,	"	88°	"	"	"	1·001

The time occupied by the fermentation varies between three and nine days. Difference of season has a great influence on the celerity or slowness of the fermentation. During the first few days the tuns are exposed as much as possible to the atmosphere; after this time they are covered over tightly to exclude the air. Two reasons have been alleged for this arrangement: first, to prevent the escape of the carbonic acid, which, it is supposed, forwards the action; and secondly, if suffered to diffuse itself into the atmosphere, it would carry off portions of the alcohol. For the first supposition some explanation may be offered. It seems that the carbonic acid, when confined, causes the greater attenuation of the liquid. It is known that the carbonic acid from fermenting tuns, when conducted through fresh worts, causes fermentation; though this is not so complete as when yeast is added. Pasteur has proved that this happens in consequence of a portion of the active principle of the ferment being carried over mechanically, and that by this the fermentation is caused; carbonic acid generated from any other source having no such effect. With regard to the second reason, it is scarcely admissible, as the fermentation is nearly terminated before shutting down the tuns.

Distillers never take yeast from the fermenting tuns, but beat it into the liquid, being of opinion that any such abstraction would render the fermentation less complete, and consequently diminish the proportion of spirit.

The best temperature for the wort, when let into the fermenting tuns, is about 70° to 75° Fahr. (21° to 24° C.); during the fermentation the temperature being at the maximum elevation, between 82° and 90° Fahr. (28° to 32° C.). Great care must be exercised to prevent the temperature exceeding 95° Fahr. (35° C.), in order to avoid acetification.

Difference in the time of the fermentation of worts is almost always owing to variation in the quality of the yeast. As the fermentation proceeds the specific gravity of the wort diminishes, in consequence of the decomposition of the saccharine matter into carbonic acid and alcohol, which is expressed by the distillers as *attenuation*, and is the standard by which they judge of the success of the operation. The amount of spirit the wash will yield may be known from the determination of the specific gravity, or degree of attenuation which has taken place.

Two reasons may be offered for the diminished specific gravity after fermentation.

1. The disappearance of the saccharine matter in the fluid. The whole of the sugar, however, is not decomposed, and consequently the specific gravity of the fermented wort would be proportionally greater than the water in its ordinary state, as the quantity of undecomposed matter is more or less.

2. The results of the fermentation are carbonic acid, which escapes, and alcohol, which remains; the alcohol, having a less specific gravity than water, diminishes the density of the latter.

The aim of the distiller is to have the spec. gr. of the wort to correspond with water. When this object is gained, it does not however follow that the whole of the saccharine matter has been decomposed. The weight of a volume of alcohol being much less than that of a corresponding volume of water, it is evident, if both be commingled, that the resulting liquid will have a specific gravity less than the latter; and to make up the strength, so as to equal the density of water, a greater or less amount of saccharine matter is requisite. In other words, a quantity of saccharine matter remains undecomposed, which raises the density considerably higher than that of water at ordinary temperatures. Generally, this quantity of undecomposed sugar increases with the original strength of the worts, as the alcohol generated in the proportionate bulk of those extracts is larger; and with it the decomposition of the saccharine matter is partially arrested, in consequence of the property which alcohol has of preventing eremacausis, or fermentation. This fact is easily proved by adding some alcohol to a portion of the wort in active fermentation, so that the liquid will indicate a specific gravity equal to, or a little below water; the action of the ferment is arrested, and the sugar, necessarily, remains in the liquid intact. If the solution be introduced into a retort, or small common copper still, and about one-third of the liquid drawn off at the temperature of the water-bath, the spent wash in the still run once more into a vessel, and a little yeast added, active decomposition of the sugar sets in, and a corresponding quantity of alcohol and carbonic acid is produced. The case is similar in the fermentation on a large scale: the annexed table shows this very clearly. The density of the nine worts examined ranged about 1·015, and the degrees of attenuation to which they were reduced were as follows:—

1.	Specific gravity after attenuation,....	1·0012
2.	" "	1·0045
3.	" "	1·0018
4.	" "	1·0000
5.	" "	1·0012
6.	" "	1·0045
7.	" "	1·0017
8.	" "	1·0007
9.	" "	1·0007

Upon examining the wash after the termination of the fermenting action, it was found that 4·34 parts of the sugar had been decomposed, and that 1 part remained intact; thus showing that when worts are even purposely diluted, nearly a fifth part of the saccharine matter will escape decomposition; and where worts are much stronger, or more concentrated, of course the loss will be much greater. On the large scale, the attenuation cannot often be carried below 1·0012. Some rare instances may occur, in which the specific gravity may be reduced to 1·000, or even 0·998; but in these instances the worts must be in a proper state of dilution, and the ferment, or yeast, of the first quality.

URE mentions the works of a Scotch distiller, where the wash when let into the tun had a specific gravity of 1·065 to 1·060. The contents of the fermenting tun were 3000 gallons of wash, and the temperature at the time of its introduction 64° to 74° Fahr. (17°·7 to 23°·3 C.). Two gallons per cent. of barm were added, and the liquid agitated. When the attenuation had reached 1·04, another gallon per cent. of barm was added.

When the fermenting tuns are small, the temperature of the worts should be higher than when the reverse is the case. Fermentation is proceeding favourably if the bubbles of carbonic acid mount in rapid succession. Should the fermentation flag, it is almost a hopeless task to restore vigorous action. Some distillers try the addition of *bubs—i.e.*, wort brought into a rapid state of fermentation in a tub by a large portion of yeast; but this plan is seldom successful. The law prohibits the addition of any wort after the expiration of twenty-four hours from the time the fermenting tun is charged, and if it be known that after that time any has been added, the distiller incurs a penalty.

With concentrated worts fermentation goes on briskly for a short time, but after the alcohol has accumulated it lags, and no further addition of yeast will revive it. The saccharine matter in such fermented worts possesses a highly disagreeable taste and smell: hence the necessity of having them properly diluted, as well as to avoid waste of saccharine matter. The worts, however, must not be too dilute, as then fermentation ceases sooner, and the diluted alcohol is more apt to run into the acetous fermentation. The attenuation is therefore urged no longer than until the head of yeast falls, the distiller thinking it more advantageous to neglect the matter remaining undecomposed, than to risk loss from the formation of vinegar. To prevent the heavy loss often incurred from this source, immediate distillation of the fermented wort is generally resorted to. Formation of vinegar is detected by the increasing specific gravity of the worts, and the peculiar aroma of acetic acid. It is, however, easily guarded against; since if atmospheric air be excluded from the fermented liquor, no acetic acid is produced. For this purpose the tuns are rendered air-tight by means of a well-fitting cover, through which a large tube passes, and enters the bottom of an open tub placed over the fermenting tun, which in this instance is filled quite full; the rising barm and froth are forced through the pipe into the open tub, and when the fermentation slackens these matters return into the tun. Many distillers use butter to keep the wash from overflowing the tun. In general, the fermenting tuns are conical vessels of much larger capacity than is required to hold the wash when first introduced, and thus serve the double purpose of containing the froth, and preventing the escape of the heat generated during the process.

At present, in the larger distilleries, the fermenting tuns are of iron, with an outer casing of wood. These are found to be much more easily managed than the wooden ones, the iron having a greater conducting power. Should the heat of the wort get too high, cold water is introduced into the space between the envelope and tun; and if it be too cold, it is readily brought to the proper temperature by supplying the jacket with hot water.

Good attenuation is the desideratum, to the attainment of which the distiller's attention is most particularly directed.

In chemistry, by the term alcoholic fermentation is understood that change which sugar undergoes under the influence of the yeast plant, and which produces wine and all other alcoholic drinks. It serves as a type of a number of other analogous phenomena known by the generic term fermentation, but specially designated by the name of the essential product of the particular phenomenon, as the panary, the lactic, the acetic fermentation, &c. The term alcoholic does not include all the phenomena of fermentation in which alcohol is produced, for many kinds have that common character; but the words are used in the sense in which they have been applied by LAVOISIER, GAY LUSSAC, THENARD, and others, viz.: to designate the fermentation of sugar with yeast.

The ordinary proposition, which is to be found at the beginning of this article, that cane sugar ($C_{12}H_{22}O_{11}$) after being modified into grape sugar ($C_6H_{12}O_6$) by absorption of water, splits up into alcohol and carbonic acid, is according to PASTEUR really only a rough approximation to the truth, although the sum of the weights of the alcohol and carbonic acid represents very nearly the weight of the sugar.

PASTEUR's researches prove that glycerine and succinic acid are also products of the alcoholic fermentation. Many methods may be used to prove the formation of succinic acid ($C_4H_6O_4$). One of the simplest is to evaporate the fermented liquid after having filtered it to separate the yeast, and then to heat the residue several times with ether, which is afterwards allowed to evaporate spontaneously. In the course of a day or two the sides of the glass are seen to be covered with crystals of succinic acid. At the bottom is a syrupy liquid full of the same crystals: this consists almost entirely of glycerine saturated with succinic acid. This process is not admissible for extracting and estimating succinic acid, but will serve to prove its presence in all fermented liquors, whatever may be their nature and origin.

Glycerine ($C_3H_8O_3$) may be detected in almost the same way, but in place of ether it is better to exhaust the residue with a mixture of alcohol and ether. This mixture dissolves the succinic acid and glycerine, and leaves behind the nitrogenized matter. The ethereal and alcoholic solution is concentrated, saturated with lime, and then evaporated to dryness. The residue is again treated with the mixture of alcohol and ether, which now only dissolves the glycerine, and leaves the succinate of lime.

When it is wished to isolate the succinic acid and glycerine completely, and determine them quantitatively, it is necessary to adopt particular precautions. The principal difficulty in the analysis of a fermented liquid proceeds from the soluble products which the yeast supplies, or which result from its transformations. The nature of these products is always the same, but their proportion varies with the quantities of yeast and sugar employed.

When yeast is mixed with a solution of sugar it gives up to that liquid a part of the soluble principles confined in the interior of its globules. Some saline matters, principally phosphates, and some nitrogenous albumenoid matters are dissolved, and the globules deriving part of their nourishment from these two sorts of substances, live, bud, and multiply. These mutations give rise to modifications of the original products, or to new bodies, some solid and insoluble, others liquid and soluble, which remain in the liquid, and are found therein when the fermentation is finished mixed with the products of the decomposition of the sugar. These general considerations must be borne in mind whilst proceeding to the separation and estimation of the products of the fermentation.

The weight of the yeast employed must first be carefully determined, and an equal weight of the same yeast must be dried at 212° Fahr. (100° C.), and then weighed, so that the amount of dry matter which the yeast contains may be known.

When, on attentively examining the fermenting liquid for some minutes, no bubbles of gas are seen to rise, we may conclude that the fermentation is finished, unless the alcoholic is complicated with the lactic fermentation, in which case the disengagement of gas may cease while a good deal of sugar remains undecomposed. But this is an exceptional case, which only occurs when the yeast employed is stale and contains some of the lactic ferment. In ordinary cases, with a weak solution of sugar, the fermentation is finished in from fifteen days to three weeks, but when an excess of sugar and but little yeast are employed, it may last for months or even years.

The fermented liquor is now filtered through a filter tared with another of the same paper. After drying at 212° Fahr. (100° C.), one weighing will give the weight in the dry state of the yeast deposited in the course of the fermentation. The filtered liquid is then very slowly evaporated to a small bulk, and the evaporation is finished in a vacuum. After twenty-four hours the syrupy residue is treated seven or eight times with a mixture of one pint of alcohol and one and a half pints of ether. Each washing should be filtered, although generally the insoluble matter remains as a plastic mass at the bottom of the capsule. To determine the amount of this insoluble matter it is only necessary to redissolve it in water, evaporate it in a water bath in a weighed capsule, and dry in a stove at 212° Fahr. (100° C.) until the weight is constant.

The flask containing the alcoholic ethereal solution is placed in warm water to drive off the greater part of the ether; the evaporation is then continued at a very gentle heat, and finished as before in vacuo. Pure and perfectly clear lime-water is then added, to exactly neutralize the acid. The evaporation is repeated with the same precautions, and the residue again treated with the mixture of alcohol and ether, to dissolve the glycerine. The succinate of lime remains behind in a crystalline state, contaminated with a small quantity of extractive matter, or lime-salt of an uncrystallizable acid. These impurities may be removed by digesting the succinate of lime for twenty-four hours with alcohol, which dissolves the foreign matters, and leaves the succinate sufficiently pure. It may then be collected on a tared filter, dried and weighed.

The glycerine may be weighed after having evaporated the alcoholic solution at a gentle heat, the evaporation being completed under the air-pump; where, however, it must not remain longer than two or three days, as it continues to lose weight after being deprived of water. In this way all the glycerine is obtained without loss, and it may be regarded as pure when the liquid has been fermented under the influence of a sufficient but not unnecessarily large quantity of yeast. When much more yeast than is necessary has been used, the glycerine will have a sharp bitter taste, because although the yeast itself has been removed by filtration, it contains a very small quantity of acrid matters which are soluble in the mixture of alcohol and ether. Fermentation by itself gives nothing which can render the glycerine impure when extracted as above.

One hundred grammes of sugar candy, fermented with 1·198 grammes of dry yeast, gave after the fermentation:—

Succinic acid,................	0·673 grammes.
Glycerine,....................	3·640 "
Total,............	4·313 "

That the elements of the glycerine are furnished by the sugar, and that the yeast contributes nothing to it, is proved by a comparison of the weights of the glycerine and yeast in the preceding experiment. But in this case it is not so clear with regard to succinic acid. It is possible, however, by a particular experiment, to obtain an amount of succinic acid larger than the soluble matter of the yeast employed. The succinic acid, therefore, is formed from the sugar as well as the glycerine, and the elements of the yeast take no part in the formation of these products.

Distillation.—The chief object of importance in this stage is the still; and no other article of manufacturing apparatus has undergone so much alteration.

The philosophy of distilling is, however, independent of the construction or form of the still, but rests upon the different degrees of volatility of the bodies subjected to the operation. By attention to this principle, any number of bodies of varied densities may be separated, if suitable means are adopted, the process being termed fractional distillation. The earliest alchemists knew that the more volatile a body, the less heat does it require to convert it into vapour; and *vice versâ*. By transmitting the vapour of two liquids simultaneously through a good condensing medium, the temperature of which is lower than the boiling point of the heavier, but not so low as the point at which the lighter boils, it is evident that the vapour of the heavier liquid will be condensed, while the other retains its gaseous state. This is beautifully illustrated by the subject under consideration. The temperature at which water boils is universally known to be 212° Fahr. (100° C.); alcohol boils at 173°·1 Fahr. (78°·4 C.). If a mixture of these two liquids be introduced into a retort or still, the mixture will boil at an intermediate temperature, proportionate to the quantity of each liquid present; but the alcohol being the lighter is driven over in larger quantity at first, carrying with it some aqueous vapour: as the boiling continues more water is given off, until, at the end of the operation, nothing passes over but steam. When the mixed vapours are conducted through a tube placed in water below 212° Fahr. (100° C.), but not so low as the boiling point of the mixed liquid, the water is condensed, and the alcoholic vapour remains unaffected, till the temperature of the refrigerator is lower than 173°·1 Fahr. (78°·4 C.). This is what happens in distillation with the ordinary apparatus. In the first and second volutions of the worm in the condensing tube, the aqueous portion is more or less condensed, but the spirit retains its acquired elasticity till it traverses the worm to where the temperature is below its boiling point; there it becomes liquid. In the days of the early distillers, when only the common still and worm were in operation, and the whole of the water eliminated with the alcoholic vapours was found in the receiver, it was only by repeated distillations of the first products that a concentrated spirit could be obtained; and this was effected by a great expenditure of time, fuel, and alcohol.

So soon as the attenuation of the wort has reached its lowest point it is run into the still with as little delay as possible. In the old method of working, the wash is distilled in two large retorts or stills, each of from 600 to 1200 gallons capacity, according to the size of the factory. These retorts are provided with a rotatory chain apparatus for preventing the lies from adhering to the bottom of the still, and becoming charred, in which case they would communicate a disagreeable taste to the spirit.

Previous to distillation, about 1 lb. of soap is added to every 100 gallons of the wash. When the charge of wash is 8000 gallons, the distillation is carried on as speedily as possible, and without risk of it "running foul," till about 2400 gallons are drawn off. These constitute the *low wines*, or singlings, and are very weak. The remainder of the spirituous product of the 8000 gallons is received in another vessel for a further distillation. The singlings are redistilled, or *doubled*, in the second still, and the spirit drawn off till it begins to acquire a disagreeable taste and smell. These are technically termed "the faints," and owe their peculiarity to essential oils held in solution. The faints are collected in the faints-back, and mixed with the muddy part of the first distillation, water is added, and the whole redistilled. Very weak singlings are the result, which, upon a second distillation, afford finished spirit.

Some distillers continue the first distillation as long as any alcohol comes over, and then subject the low wines to a second distillation in the spirit still. The first portions are more or less blue or muddy, and consequently are run into the faints-back. As soon as the spirit becomes clear, and devoid of a disagreeable odour, it is run into the spirit-back. The quantity of the spirit obtained in well-regulated distilleries amounts to about three-fourths or even four-fifths of the low wines operated upon; faints are drawn over at the end of the distillation, and are turned into the faints-back, together with the first portions. These faints are mixed, as before stated, with a considerable quantity of water, and distilled, in order to free them from the disagreeable oil eviscerated by the husks of the grain.

A self-regulating bath is, in some distilleries, put in the capital of the still. The common Scotch stills have the capital 15 to 20 feet high, to prevent the wash from boiling over into the worm; it is customary to strike the capital from time to time with a rod, and from the sound emitted, it is inferred whether it be empty, partially filled, or in danger of an overflow; in the latter case the fire is withdrawn or damped by means of a spout near the furnace-door, and which is supplied with water from a cistern in the upper part of the building. When a very pure spirit is required, it is customary to dilute the liquor with water and submit it to a third distillation, in order that the distillate may not have the harsh taste of strong alcoholic liquids. By the use of the improved stills, a liquid 60 per cent. over proof is obtained, even in the first distillation, and at a considerable saving of fuel, time, and labour, while the use of soap, &c., is unnecessary.

The usual yield of proof spirit from malt is between 2 and 2½ gallons per bushel. The largest amount of spirit procured from one quarter of corn is 20 gallons. As a general rule, the lower the heat at which the distillation is carried on, the purer will be the spirit. When an excess of soap has been used, and the distillation urged too rapidly, the distillate often possesses a saponaceous flavour, which is occasioned by its fatty particles being carried over mechanically in the vapour, and dissolved in the alcoholic liquid. The manner in which the soap acts to prevent the charge running foul is as follows:—During fermentation and subsequent transference of the wort into the still, small portions of acetic acid are generated, which decompose part of the soap, setting free the oily compound, which rises to the surface of the liquor, and breaks the bubbles of vapour as they ascend through it from the bottom of the retort; hence the liquid cannot pass over unless the boiling be violently urged.

The average quantity of spirit obtained by the Irish distillers from a barrel of malt, *i.e.*, 12 stones, is 8½ gallons, Irish measure, or 6 gallons 5 pints and 1½ noggins, imperial measure, of 24 per cent. over proof.

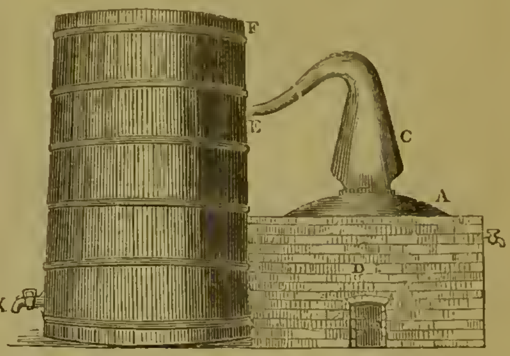
Dr. URE performed several experiments, at the request of the Board of Excise, for the purpose of deciding a discussion which had taken place in Ireland relative to the extent to which raw grain could be fermented; the most decisive of his results is the following:—3 bushels of mixed grains were taken, consisting of 2 of barley, one-half of oats, and one-half of malt, which, being coarsely ground by a hand-mill, were mashed in a new tun with 24 gallons of water at 155° Fahr. (68°·3 C.). The mash liquor drawn off amounted to 18 gallons, at the density of 1·0465; temperature of 82° Fahr. (27°·7 C.). Being set in a new tun, it began to ferment in the course of twelve hours, and in four days it was attenuated down to spec. grav. 1·012. This yielded, upon distillation, 3·22 gallons of low wines, and after rectification, 3·05 gallons of spirits, the quantity equivalent to the attenuation by the tables being 3·31.

Before the fermented wort goes into the still, a calculation is made by the excise officer on duty of the quantity of wash drawn from the wash-back, which has been pumped into what is called the wash-charger. If the liquor in the charger exceeds the quantity in the back, the distiller has to pay on the higher amount; if it contain less, he must pay according to the wash-back, as being the larger quantity. When all the wash is transferred to the charger, its exit tap is unlocked, and the wash is allowed to be drawn off into the still; the charging and discharging tap of the still being fastened by the officers, so that there can be no transfer of wash except through the pumps. The first distillation from the wash is worked into the low-wine receiver, and the strength and quantity are ascertained by the excise officer and compared with the quantity which the contents of the wash-back had been estimated to produce. This is then pumped from the receiver into the low-wine charger, and after the officer has performed his duty, it is permitted to be drawn off into the low-wine still, which is a distillation of the second extraction; the low-wine still then works into another cask, called the spirit-receiver; when that distillation is finished, the officer, reattending on regular notice for that purpose, takes the quantity and strength of the spirit therein, and upon the quantity so ascertained he charges the duty. If it happens that the actual quantity of spirit, after the distillation, differs from the hypothetical quantity ascertained by previous calculations, he gives to the government the benefit of the doubt, and levies duty on the higher quantity, whichsoever that may be.

In distilling low wines, one portion of them goes into the spirit, and another into the faints-receiver; these faints are, in the next distillation, united with the low-wines from the succeeding wash-back, and are worked together; the united produce goes partly into the spirit-cask, and partly back into the faints-cask. The operation is thus continued till the backs are emptied. All these backs, chargers, receivers, &c., are secured by locks, the keys of which are kept by the excise officer.

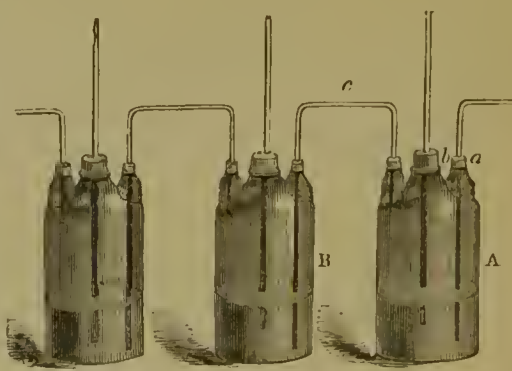
Fig. 6 is a drawing of the common still: A is the body incased in brickwork, D, and directly over the fire: C, the head attached to the condensing-worm, E, placed in the water-tub, F, where the vapour is condensed to a liquid which flows into a receiver at K. The still is of variable dimensions, from 10 to 500 gallons. This form of apparatus was in use for a considerable period.

Fig. 6.



WOULFE, having the principle of GLAUBER'S apparatus in view, was the first to apply the facts already cited, in order to obtain strong spirit, and various other products of distillation. His apparatus bears the name of its inventor to the present day. It is shown in Fig. 7, and consists of a series of bottles, placed in a range. Each of these bottles has three openings or necks; the first bottle was connected with the beak of the still by a pipe or tube, which passes through the neck, *a*, to within an inch or two of the bottom. A safety-tube is introduced through the neck, *b*, and dips into the liquid in the bottle. The bottle, A, communicates with the bottle, B, by means of a pipe or tube, *c*, bent at right angles; in the first it opens a little below the cork, but in the second it passes nearly to

Fig. 7.



the bottom. The second is joined in a similar way to a third and fourth bottle, if required. This apparatus is in constant use in the laboratory, for the purpose of obtaining aqueous solutions of gaseous bodies. The same principle is to be observed in all the various modifications of stills constructed in the beginning of the present century, the most important of which will be described.

In this kingdom, the modifications in the construction of stills have been various. Previous to the year 1788 the old form of still was in general use. From the slowness of the distillation, a week elapsed before a charge was completely worked off, and even then the products were very dilute. At this period the excise duty levied was according to the size of the still, and no further trouble was taken by the officers as to how the worts, &c., were made, except that they visited the distilleries occasionally, to observe if any other stills were in operation, or if larger ones were substituted for those which had been already gauged. About the above period an important revolution took place in the construction of this apparatus by a firm in Leith, by which the distillation was very much expedited. They lessened the height and increased the width of the still, to expose a larger surface to the action of the fire than could be done in the old form; the head of the still was enlarged in proportion to the quantity of vapour generated, and occasionally several outlets or pipes were inserted around the horizontal upper part, to facilitate the escape of the steam and alcoholic vapour into the condensing worm. This still could be charged, distilled off, and be ready for another operation, in the course of a few hours. The inventors preserved to themselves its exclusive use for about twelve months; but such an important discovery could not escape the vigilance of competing neighbours, and hence its use shortly afterwards became general throughout Scotland. The excise, until they became apprised of the fact, were outwitted, the distillers evading the duty which they should have paid for the excess of spirits distilled above the allowance to which the former method of gauging subjected them.

Fig. 8.

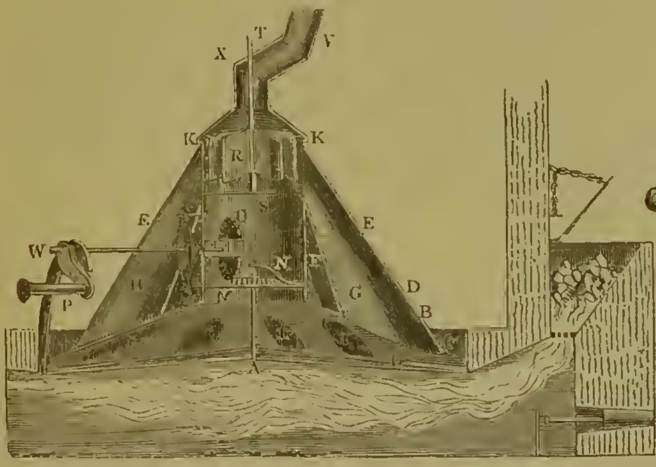
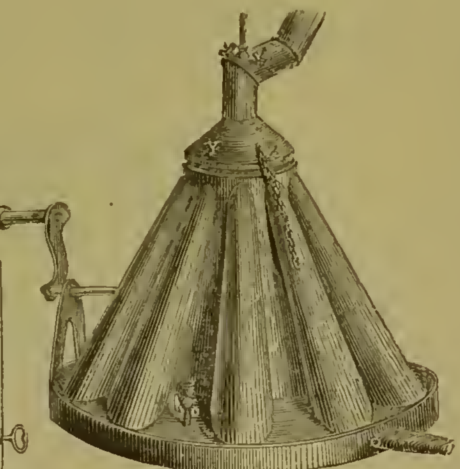


Fig 9.

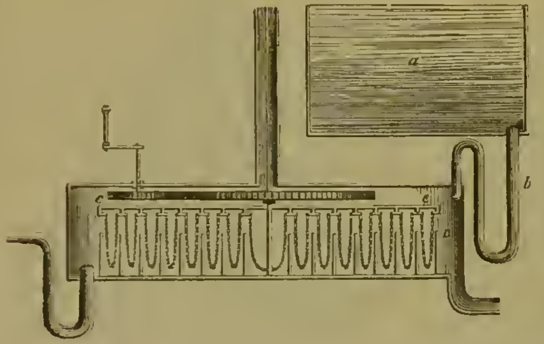


The excise duty, however, was upon this soon altered, and year by year increased; but the distillers, constantly upon the alert, still cheated the overseers appointed by Parliament. At length, in 1799, a parliamentary committee was appointed to investigate this branch of the excise laws. In consequence of their report the distillers were subjected to an excise duty according to the capacity of the still, and on the supposition that it would be worked off and charged every *eight* successive minutes during the distilling season. Even this time was considerably shortened by the distiller; but the amount of fuel consumed, and the consequent wear and tear, left it a matter of doubt whether with any profit. The rapidity of this distillation was carried so far, that in 1815, the last year of the license duty, a still of 80 gallons capacity could be distilled off, emptied, and be ready for a successive operation in three and a half minutes, sometimes in three minutes! A still of 40 gallons could be drawn off in two and a half minutes. An alteration in the excise laws at this time did away with the license duty, and the law became the same as in England, the duty being levied upon the wash and spirits procured therefrom.

For a long time, however, Scotch stills were constructed on the plan of those in use during the period of rapid distillation, namely, by having their bottoms wider than the English stills, in proportion to their size.

The subjoined Figs., 8 and 9, represent a sectional and front view of the Scotch still, at the period when rapid distillation was popular amongst spirit manufacturers. E E is the body of the still, the bottom of which is about 16 feet in diameter, and convex towards the middle; the depth of the still at the centre is about 11 feet, and the sides and bottom meet at an acute angle at the verge. The hollow bottom of the still is connected by solder or rivets with the shoulder, B; C is a rim, which serves to support the still and also to protect it from the fire. D, seen in the front view of the apparatus, is the discharging pipe. In the shoulder of the still are several elliptical openings, from which proceed oblique conical tubes, which enter the cylinder rising from the centre of the boiler; G and H are two of these pipes; I I I I are lower openings; and K K the top openings; F is a section of the central column; L, an agitator within the still, to keep the bottom free from sedimentary matters—a chain agitator was sometimes substituted; M, a vertical axis of the agitator, to which a horizontal toothed wheel, N, is attached; O is another such cogged wheel, but vertical, gearing into the wheel, N, so as to communicate its motion to the latter when required; P, a handle fixed upon the axis of the wheel, O, by which it is turned; W, support of handle and axis; R, a fan to break the froth formed by boiling; its axis rests upon the cross-bar, S. Motion is given to the fan by the vertical axis, T; this axis enters the large pipe that carries off the vapours, through a steam-proof packed box, X; a similar box surrounds the axis of the wheel, O; V is the pipe communicating with the condensing-worm, through which the vapours escape to be condensed.

Fig 10.



The funnel pipe, O, shown in the perspective view, serves to charge the still, and Y is the cover of the central column, which is held in its proper position by chains, as seen at Z.

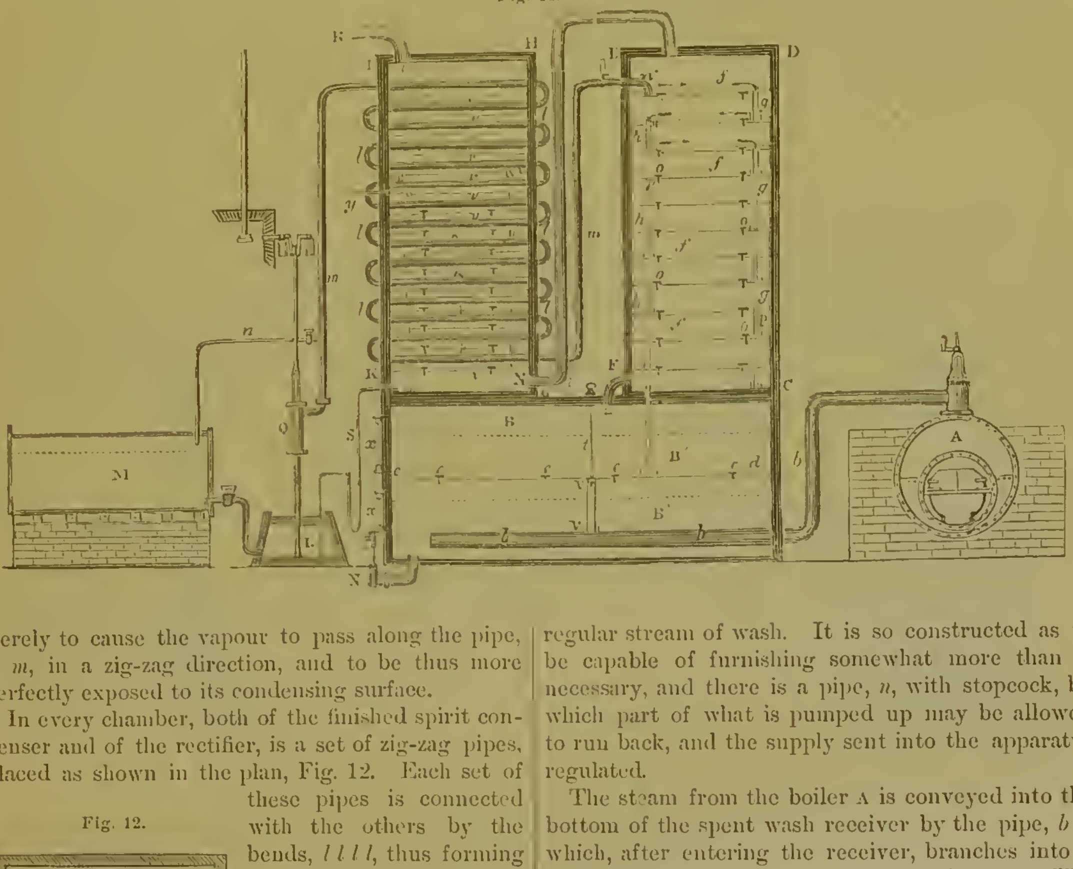
An adaptation of this still, by Sir Anthony Perrier of Cork, is shown in Fig. 10. The liquid to be distilled is made to flow gradually and continuously over the heated surface of the boiler, while it parts with its alcohol. The bottom of the boiler is divided by concentric partitions, sufficiently high to prevent the liquid from boiling over; these partitions have openings from one to another at opposite sides, so as to make the course a sort of labyrinth. *a* is the reservoir of liquor prepared for operation; *b*, a pipe descending from this reservoir, which conducts the liquor into the boiler at *c*, the commencement of the labyrinth, in flowing through which it progressively traverses the whole surface of the bottom, so that the full effect of the fire is exerted upon thin layers of the liquid. This causes the evaporation to proceed with great celerity, and when the liquid reaches the discharge-pipe at the opposite side, it contains no alcohol. The series of chains suspended from the bars, *e e*, which are supported by the central shaft, prevent the deposit of mucus and albuminous matters on the bottom of the still. A toothed wheel and pinion communicate motion to the bars, *e e*, through the shaft, and the chains sweep the compartments between the partitions as they turn round.

The first step towards improving the condensing apparatus was taken by Coffey of Dublin. In all the preceding stills, though great rapidity of distillation had been attained, yet the great disadvantage remained of having the aqueous and alcoholic portions of the distillate mixed up, so that it was only by repeated distillations that a strong spirit could be obtained. Coffey inserted two pipes in the first and second rounds of the condensing worm, and led them back to the body of the still. By this simple contrivance a great part of the water was removed from the mixed alcohol and aqueous vapours by being condensed in the first convolutions, and returned to the still instead of flowing into the receiver with the spirit.

Subsequently Coffey patented another still, which gives in continuous distillation the strongest spirit that can be obtained on the large scale. Fig. 11 is a section of this still. The body of the apparatus consists of an oblong vessel, B, and two columns erected thereon, C D E F and G H I K. The first of these columns is called the analyzer, the second the rectifier. The whole is made of wood, lined with copper, and the wood being 5 or 6 inches thick, little or no heat is lost by radiation. The oblong vessel has a copper plate or diaphragm, *c d*, across the middle of it, which divides it into two chambers, B′ B″. This diaphragm is perforated with a great number of small holes, for the passage of the vapour upwards, and it is also furnished with several valves, which open upwards, as shown at *e e e e*, whenever the vapour is in too great quantity to find a free passage through the perforations. A pipe, V V, fitted with a valve, which can be opened or shut at pleasure by means of a rod, *t*, passing through a stuffing-box on the top of the vessel, descends from this diaphragm nearly to the bottom of the lower chamber, into a pan forming a steam trap. Glass tubes, at *x x*, show at all times the level of the liquor in the chambers, B′ B″. The column, C D E F, which is called the analyzer, consists of twelve chambers, *f f f f*, formed by the interposition of eleven copper diaphragms, marked *g h*, which are perforated with very numerous holes, and furnished with valves, *o o o o*, opening upwards. To each of them is also attached a dropping pipe, *p*, by which the liquor flows from plate to plate; the upper end of each pipe projects an inch or two above the plate in which it is inserted, so that at all times, during the distillation, a stratum of wash of that depth remains upon each diaphragm. The lower end of each pipe dips into a shallow pan on the diaphragm beneath, thus forming a steam trap, by which the escape of vapour through the pipe is prevented. The pipes are inserted at alternate ends of the diaphragms, as shown in the figure.

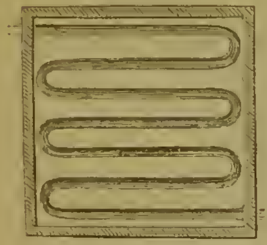
The column, G H I K, is divided into chambers by interposed copper plates, in a similar manner to that just described. There are fifteen chambers in this column, the lower ten, *k k k*, &c., constitute the rectifier, and its diaphragms are perforated and furnished with valves and dropping pipes, precisely similar to those of the analyzer. The upper five form the finished spirit condenser, and are separated from the other ten by a copper diaphragm, without perforations, but having a large opening at W, for the passage of alcoholic vapour, and a dropping pipe at *s*. Round the opening, W, is a neck, rising an inch or so above the surface of the diaphragm, which prevents the return of any finished spirit by that opening. Under the dropping pipe, *s*, is a pan much deeper than those of the other dropping pipes, and from this pan a branch pipe, *y*, passes out of the apparatus and carries the condensed, but still very hot spirits, to a worm, or other refrigerator, wherein they are cooled. The chambers, *v v v v v*, of this spirit condenser are formed of plain unperforated diaphragms of copper, with alternate openings at the ends, large enough both for the passage of the vapour upwards and of the condensed spirit downwards; the use of these diaphragms being merely to cause the vapour to pass along the pipe, *m m*, in a zig-zag direction, and to be thus more perfectly exposed to its condensing surface.

Fig. 11.



In every chamber, both of the finished spirit condenser and of the rectifier, is a set of zig-zag pipes, placed as shown in the plan, Fig. 12. Each set of these pipes is connected with the others by the bends, *l l l*, thus forming one continued pipe, *m m*, leading from the washpump, Q, to the bottom of the rectifier, whence it finally passes out, and rising up, enters the top chamber of the analyzer, where it discharges itself at *n′*. M is the wash charger; L, a smaller wash vessel connected with it and with the wash pump. This vessel is called the wash reservoir; its use is to retain a sufficient reserve of wash, to prevent the apparatus being idle during the delay, which the excise regulations render unavoidable, between the emptying of the wash charger and the refilling it from a new back.

Fig. 12.



The pump, Q, is worked continuously during the distillation, so as to supply the apparatus with a regular stream of wash. It is so constructed as to be capable of furnishing somewhat more than is necessary, and there is a pipe, *n*, with stopcock, by which part of what is pumped up may be allowed to run back, and the supply sent into the apparatus regulated.

The steam from the boiler A is conveyed into the bottom of the spent wash receiver by the pipe, *b b*, which, after entering the receiver, branches into a number of small pipes, perforated with holes. The steam is thus dispersed through every part of the wash in which the pipes are immersed. These perforated pipes are not shown in the drawing.

When commencing an operation the wash pump is set in motion to charge all the zig-zag pipes, *m m m*, until the wash passes over into the analyzers at *n′*. The pump is then stopped, and the steam let into the bottom of the apparatus by the pipe, *b b*. The steam passes up through the chambers, B″ B′, and by the pipe, *z*, into the analyzers, whence it descends, through *i*, to the bottom of the rectifier at N. It then rises through the chambers, *k k*, enveloping the zig-zag pipes, and rapidly heating the wash contained in them. When the attendant perceives, by feeling the bends, *l l*, that the wash has been heated in several layers of these pipes, perhaps eight or ten layers (the number is not of much moment), he again sets the pump to work, and the wash, now nearly boiling hot, and always in rapid motion, flows from the pipe *m* at *n*′, and passes down from chamber to chamber through the dropping pipes, in the direction shown by the arrows in a few of the upper chambers. It may be here observed that no portion of the wash passes through the small holes perforated in the diaphragms which separate the chambers. These holes are regulated, both in number and size, so as not to be more than sufficient to afford a passage for the vapours upwards, when under some pressure. The holes, therefore, afford no outlet for the liquor, which can only find its way down in the zig-zag course indicated by the arrows. It is therefore obvious that the wash, as it passes down, is spread into as many strata as there are diaphragms, and is thus exposed to the most searching action of the steam constantly blowing up through it. As it falls from chamber to chamber its alcohol is volatilized by the steam passing upwards; and by the time the wash has reached the large chamber, B, no trace of the spirit remains. The wash, as it descends from the analyzer, accumulates in the large chamber, B′. When this vessel is nearly filled, which can be perceived by inspection of the glass tube, the attendant opens the valve of the pipe, V, and discharges the contents of B′ into the lower compartment: then shutting the valve, the wash from the analyzer again accumulates in B′, and when it is nearly full, the contents of the lower chamber are discharged from the apparatus altogether, through the cock, N′, and the charge in B′ let down by opening the valve, V, as before; thus the process goes on so long as there is any wash to supply the pump.

When all the wash is gone, water is let into the reservoir, L, and pumped through the pumps, *m m*, to obtain the last portions of alcohol.

It has been already said, that in the ordinary course of the operation the wash is stripped of all its alcohol by the time it has reached the bottom of the analyzer; but as a precautionary measure the chambers, B′ B″, have been superadded, in each of which the spent wash is exposed for about half an hour to the action of the steam blowing through it. There is a small apparatus—not shown in the engraving—by which a portion of the steam in the chamber, B″, is condensed, cooled, and made to flow constantly through a sample jar, in which is a hydrometer, or what is better, two glass bulbs, one of the spec. grav. 1·00, and the other, 0·998. The attendant knows all is right when the lighter of these bulbs floats in the sample; hence the chamber, B, may be emptied without any risk of loss.

The course of the wash being understood, that of the steam requires very little description.

The steam is first blown through the charges of spent wash in the lower chamber of B, thence it passes up through the layers of wash on the eleven diaphragms of the analyzer. In its course it abstracts alcohol from these layers of wash, depositing water in its place. After traversing the whole of the analyzer, the vapour, now containing much alcohol, passes by the pipe, *i*, into the bottom of the rectifier, and as it ascends it envelopes the pipes *m m*, heating the wash, and simultaneously parting with its more watery portion, which is condensed, and falls in a state of ebullition on the several diaphragms of the rectifier. By the time the vapour reaches the passage, W, in the bottom of the finished spirit condenser, it is nearly pure alcohol; and as it is condensed by the wash in the pipes, and falls on the diaphragm, it is conveyed away by the pipe, *y*, to a refrigerator. At the top of the spirit condenser is a large pipe, R, which serves as a vent for the uncondensable gas disengaged in the process, and this pipe also communicates with the refrigerator, so that should alcoholic vapour at any time pass out of the apparatus, no loss is sustained beyond the waste of fuel caused by condensing it by the water of the refrigerator, instead of the wash of the condenser.

The liquor formed on the several diaphragms of the rectifier descends to the bottom in the same manner as the wash falls from chamber to chamber in the analyzer; but as it still contains alcohol, it is conveyed by the pipe, S, to the pump, Q, by which it is raised up with the wash to be again distilled.

A thermometer at *m*′ shows the attendant the temperature of the wash as it issues from the pipe, *m m*, into the analyzer, being the only guide he requires for managing the operation: for when the temperature is what it should be, nothing can go wrong in the work. Whenever the thermometer indicates too high a temperature, more wash is let into the apparatus, and *vice versâ*—the quantity being regulated by the tap and the pipe, *n*. Very little nicety is requisite on this point, the fluctuation of a few degrees above or below the proper heat is of little consequence, and it is very seldom found necessary to alter the supply of wash.

The water for supplying the boiler passes through a long coil of pipe immersed in boiling-hot spent wash, by which means it is raised to a high temperature before it reaches the boiler.

The vapour passing through this apparatus is condensed by the wash, and not by water; and no heat is wasted as in the common process.

The continental savans at an early date improved the forms of stills. Poissonier, in the year 1779, proposed a modification of the common still, which would probably have come into general use, had it not been for the better and more ingenious invention of M. Adam, before alluded to. Being an auditor of a course of chemical lectures at Montpellier, during which the merits of Woulfe's apparatus as a condenser was discussed, Adam conceived the idea of constructing a still in which the principles of the apparatus of Glauber and Woulfe should be applied to the condensation of alcoholic vapour. In 1801 he took a ten years' *brevet*, or patent, for his invention, and since that time a complete revolution has been effected in the art of distillation. About the same time Solimani obtained a patent for another form of distillatory apparatus. This gentleman was a physician at Nimes, and formerly lecturer on chemistry and experimental philosophy, and disputed the priority of his invention with ADAM; but his patent is dated July, 1801, a few days later than ADAM'S. Various other modifications of the still and condensing apparatus were shortly after introduced, the most important being that of M. BERARD, patented on the 16th of August, 1805.

Fig. 13 is a drawing of ADAM'S still; B is the body of the still, incased in brickwork, and heated by the furnace, A. The head, I, of the still carries off the alcoholic vapour to a series of egg-shaped copper vessels, H, H, H, the first of which it enters at the top, and terminates at the bottom in a perforated rose, like that of a watering-pot, the holes being about an eighth of an inch in diameter. The first vessel is connected with the second, and the second with the third, by the pipes, K, M, proceeding from the top of each, and terminating at the bottom of the next, in the form of a rose, similar to the pipe, I, the tubes fitting air-tight into them. They are supported on a framework, Q P, the wider end being uppermost. D, D, D are cocks, to show when they are half-full. The last vessel—here the third—is furnished with a bucket, N, soldered to its upper end, and filled with water to condense the vapour; the hot water is drawn off by the stopcock, O. When the still is furnished with four or more oval-shaped vessels, the last two have refrigerators attached to them; if strong spirit be not required, the third may be dispensed with. The pipes, S, R, furnished with stopcocks, connect the second and third vessel with the globe, T, from which the worm in the covered vessel, U, proceeds. V is a large tube, which contains the second worm, being a continuation of the one in the vessel, U, and is filled with cold water by means of a water-pipe, entering at the bottom, though not shown in the figure; and as the water gets warm, it is discharged by the pipe, *c*. Another pipe, *a b*, issues from the head of the vessel, U, and is inserted in the globe, T, a continuation of which, though not shown in the figure, connects this globe with the body of the still, or with either of the egg-shaped vessels, at pleasure; *g g g* connects the vessels U with the body of the still, as also with H, H, H, by means of the branching pipes from their bottoms, which are furnished with stopcocks, *h*, *i*, *k*; the stopcocks, *l*, *l*, *m*, *n*, in the pipe *g g g*, serve to regulate the connection with either of these vessels, as occasion requires it. The pipe and stopcock in the shoulder of the boiler regulate the proper quantity of wine to be introduced; and C, another pipe and stopcock, serves to run off the *vinasse*, or spent wine, from the still, when all the spirit has been eliminated. Another pipe, X X X, connects the three vessels, as well as the capital of the still, with a small worm placed in the vessel, F, the connecting branch pipes being furnished with stopcocks, to open or close a connection with any of the vessels. O O is a funnel pipe, which serves to charge the apparatus with *repasse*, or weak brandy, and is joined with the first, H, and the frame, Q P, by iron stays. The whole of the still and condensing apparatus is constructed of tinned copper, and the pipes connected by solder.

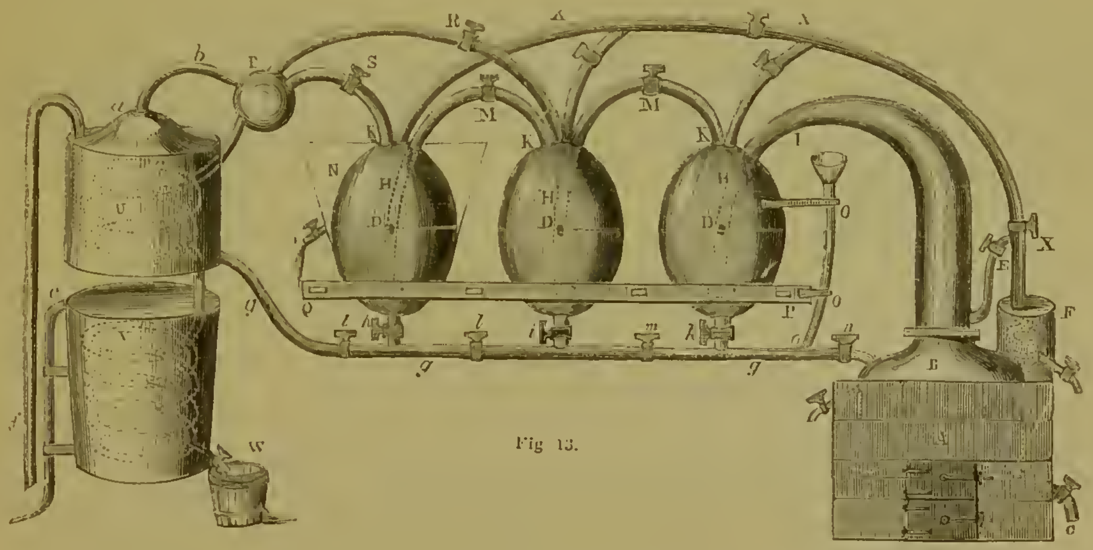


Fig 13.

Distillation is begun by opening the stopcocks, *l*, *l*, *m*, *n*; also, the pipe in the shoulder of the boiler, and closing the cocks, *h*, *i*, *k*; the vessel, U, is then filled with wine through the supply pipe, *f*, inserted in the cover, by means of a forcing pump, till the wine flows out by the pipe in the shoulder of B; this is then closed, and the cocks, *k*, *i*, *h*, opened in succession, till the wine flows out at D D D, the stopcocks, *n*, *m*, *l*, being closed as the still and first and second vessels are filled in succession. The pumping of the wine is continued until the vessel, U, is nearly filled; the refrigerators, N and V, are then filled with cold water. Everything being thus prepared, all the lower cocks are closed, and the upper stopcocks, M, M, R, S, opened, in order to allow a free passage for the vapour; the fire is then urged on till the liquor in the still begins to boil. The first portion of vapour is richest in spirit, and this passing into the first vessel H, by the capital I, is condensed. The wine in this vessel—which is heated by the vapour rising from the boiler—contains a greater proportion of alcohol, on which account it boils quicker, and cannot reach so high a temperature as the liquid in B. The eliminated vapour from the first H, in consequence of the low temperature at which it is generated, contains less water, and this being condensed in the second, renders the wine which it contains much stronger than that in the first. It consequently boils at a still lower heat. The more aqueous portion of the vapour from the second H, passing off by the connecting pipe, K M, is partly condensed in the third, while the concentrated alcoholic vapour enters the condensing worm in the vessel, U, through the pipe, S, where it is condensed, and flows out into the receiver, W. This receiver is tightly covered to prevent evaporation of the alcohol; and in order that the amount of liquid undergoing condensation may be seen, a glass pipe connects the lower end of the worm and the receiver. As soon as the distilled product, when examined by the hydrometer, shows a diminution of strength, the receiver is changed, and the weaker liquor, or *repasse*, is submitted to a second distillation. The strength of the liquor in the boiler, B, or in any of the condensers, may be ascertained by means of the pipe, X X X, which proceeds from the last, and communicates with the small worm, F, beside the body of the still; from each of the others, as also from the capital, pipes open into it, and as these are each furnished with stopcocks, the vapours from any particular condenser, or the still, may be liquefied in the small worm, F, the other communications being cut off. The spirit as it flows out of the end of this worm is received in a testing glass, and examined by the hydrometer, or by other means. When the liquor in the body of the still is exhausted, the fire is withdrawn, the other communications with the oval-shaped vessels and great worm are cut off, and the cock, C, opened to draw off the *vinasse*, or residue. If it appears that the liquid in any of the condensers is exhausted, it is run off to the body of the still by turning the stopcocks appended to it. The still is next charged by allowing the unexhausted liquor in H, H, H, to flow in through the pipe *g g g*, and the remainder, sufficient to fill the boiler as before, is supplied from the vessel, U. H, H, H may be half filled with brandy, or *repasse*, through the funnel pipe, O O; after which the lower taps are closed, and the upper ones opened as before, and the distillation continued. During the transmission of the alcoholic vapour through the wine vessel, U, the contents become heated, and some spirituous vapour is given off, which may be conducted into the body of the still, or any of the condensers, as deemed desirable, by the pipe *b*, the continuation of which is not expressed in the drawing.

When weak spirit is required, the communication with the third H is cut off by closing the stopcocks, M, S, and opening R, and when an extra strong liquor is required, a fourth condenser is supplied; for the greater the number of condensers, the better and more completely will the rectification be effected. The body of this kind of still is required to be stronger than ordinary, in consequence of the pressure from the Woulffe's bottles, H H H, which, of course, renders the expansive force of the vapour greater.

An elevation of the apparatus of Solimani is represented in Fig. 14, of which Fig. 15 is a section. The distillation is effected by the heat of boiling water. Four stills constitute the set; these are A, A, B, B, in the section, two are placed at each side of the chimney, T. Each pair of stills is connected at the bottom by the pipes, E, E′; the body of each still is about 4 feet square and 18 inches deep, and rests upon stout iron bars, *c c c*, firmly fixed in the walls of the furnace. *d d d′ d′* are the necks of the stills; they measure about 3 feet in breadth, and are long enough to rise above the stone vault, *f*. The heads of the stills are rather low, and their curvature, *g g g′ g′*, rather wide; they are soldered to a large pipe, H H′, which conducts the alcoholic vapour to the copper vessels, K K′, forming a part of the condensing apparatus. Some of the vapour is condensed in these vessels, and forms a liquid layer on the bottom, through which the remaining vapour has to force its way. F F′ are large pipes for carrying off the uncondensed gas into the vessels, I I′, wherein is contained the condensing apparatus, or "dephlegmator." Another small pipe, not shown in the figure, carries off the vapour from I I′ into the condenser, which is immersed in water in the stone cistern, M; and the small pipes, *a a′*, supply the cold water to the vessels I I′ from M.

The stills are charged with wine through the main pipe, P, which branches into them. By turning the stopcocks of this pipe, either pair of stills may be charged with wine, as the pipe, *e*, and the termination of P, reach nearly to the bottom of the body of the stills. The pipe, X, X, supplies the large cistern, M, with cold water.

The condenser in each of the vessels, I I′, is kept cool by a self-acting apparatus, that admits the water from the cistern, M, in the requisite proportion to cool the parallelogram condenser, Fig. 16.

The stills are heated by two boilers, about 10 feet long and 4½ wide, which contain a depth of about 8 to 12 inches of water. *b b b′ b′* show a portion of the flue, which, at the fire, is about 8 inches square, and gradually gets narrower as it approaches the chimney. The length of the flue from the fire to the chimney is about 36 feet, being brought several times back and forth under the boilers, that no heat may be lost. *z z′* are pipes for carrying off the steam into the chimney.

During the time the stills are in the course of being charged, the water in the boilers is raised to the boiling point, and the steam which is generated, circulating around the stills, quickly raises them to the requisite temperature. The alcoholic vapour passes through H H′ into the vessels, K K′; part of it is here condensed, the remainder enters the first condenser through the large pipe, F F′. All the condensed liquid that is formed in this refrigerator flows back through the pipes, F F′, into K K′, where it collects till it rises as high as the bend of the

Fig. 14.

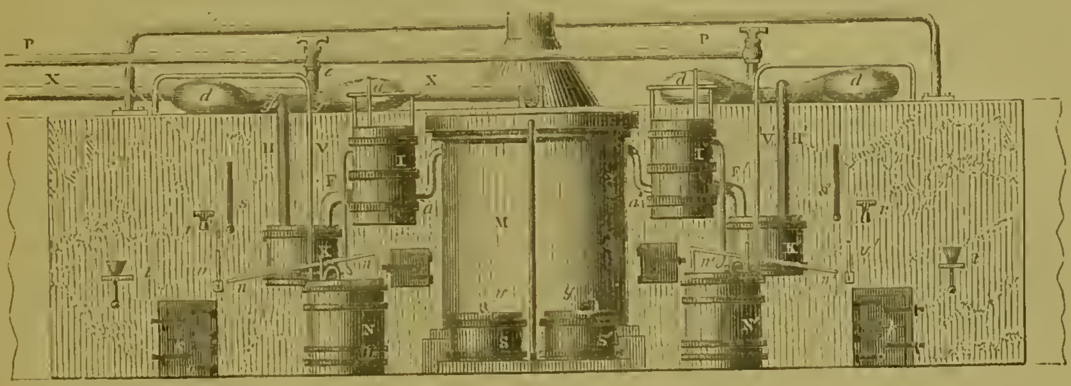
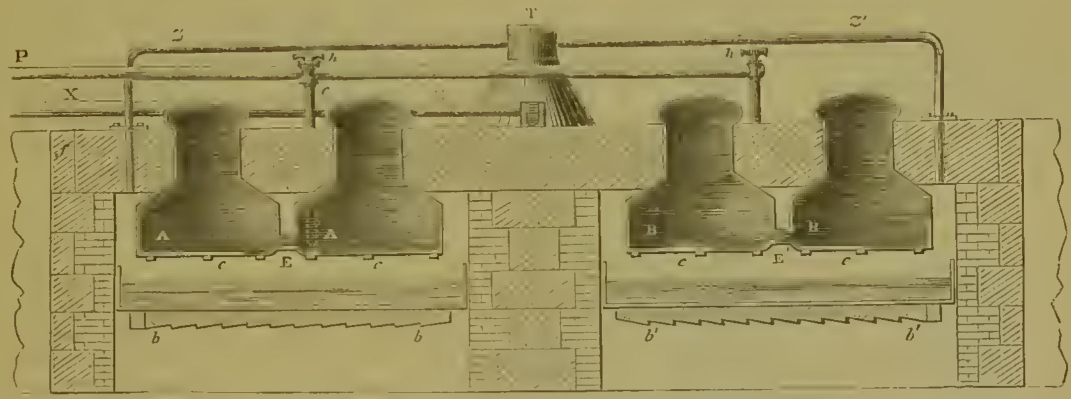


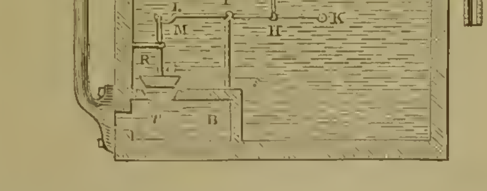
Fig. 15.



attached siphons, when it flows into the large covered tanks, x x′. When x is full, it is pumped into the stills by means of the pumps, *n n n′ n′*, through the pipes, v v′, to undergo a second distillation. *i i′* are doors, by which to enter when any repairs are required by the boilers, &c.; *k k′*, the doors of the furnaces for heating the boilers, and *o o′*, the ashpits. The pipes which discharge the spent wine from the stills are seen at *r r′*, and the gauge, or glass tube, *s s′*, shows what height of liquid they contain. The funnel pipes, *t t′*, serve to introduce water into the boilers, which are furnished, like the stills, with glass gauges, *v v′*, to show the height of water inside.

Fig. 16 is a section of one of the vessels, I, on one side of the condensing apparatus. B is a box fixed on the bottom, having a valve, C, in its upper part, of sufficient weight to resist the force of the stream of water entering the box by the pipe, *l*, from the large condensing trough. E F is a floating ball, bearing on its upper stem, G F, a basin, G, for the reception of weights. The lower stem of the float has a ring at the end, H, through which a sliding rod, K L, passes; this rod has a weight at K, and a hook at L, which passes through the ring in the upper end of the rod, M, attached to the valve, C; K L is supported in the centre by the upright, P; R is a horizontal rod which retains the valve, C, in its proper position, by means of the stem of the latter passing through a ring at the end of R. A sliding rod, S T, supported by the arm, Q, is inserted in the side of the vessel; this rod has a ring, *i*, at its end, through which the upper stem of the float, E F, passes. The principle of the working depends on the expansion of the water when heated, and its proportionate decrease of specific gravity. Sufficient weight is placed on the basin, G, to counterpoise the float at the exact temperature at which it is desirable to have the water. When the water gets hotter

Fig. 16.



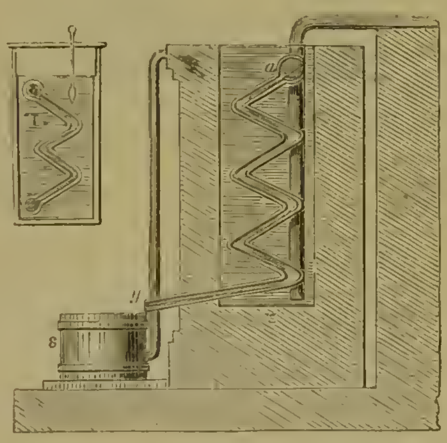
than this, the float descends, and pressing on the lever rod, K L, at H, raises the valve, C, upon which the water from the pipe, *l*, enters; when sufficient water has entered to cool down the vessel to the proper degree, the water increases in density, and the float again rises and closes its valve.

When the float does not act with sufficient promptness, the rod, S T, is pushed further into the vessel, which moves the float towards K, increasing the leverage by which it acts on C, and by this means greater force is applied to raise the valve C, in order that the cold water may enter.

The condensing apparatus, or dephlegmator, in the vessels I I′ and M, in Fig. 14, consists of two broad sheets of tinned copper soldered together, so closely as to leave only one-sixth of an inch between them. In the vessels I I′, the dephlegmator forms four inclined planes, and in M M′ it is composed of six. These are the more advantageous on account of the extent of surface which is exposed to the condensing action of the strata of water. Figs. 17 and 18 show the position of these condensers.

Having described the still and condensing apparatus, the manner of working will now be shown. The boilers being replenished with water to the depth of about 8 inches, the fire is urged under them; during the time this is being done, wine runs into the still through P and *e* (Fig. 14), till it reaches the proper height, as indicated by the gauge *s*, when the taps of the supply pipe are turned off. As soon as the water begins to boil, the contents of the still are likewise heated, and the vapour produced is forced to descend by the pipe, H, into the vessel, K, where some of the aqueous portions are condensed. The remaining portion of the vapour traverses the layer of liquid in its passage into the condensing vessel, I. Here the excess of steam is condensed, and flows back into the vessel, K, through the pipe, F; and when the weak and impure spirit collects in this to the height of the bent siphon tube, it is discharged into the tank, N, whence it is again returned to the still by the pump, *n n*. But very little spirit is condensed in I I′, when the apparatus in this vessel for the regulation of the supply of cold water is properly attended to, and the strong alcoholic vapours pass off into the condensing vessel, M, where they are liquefied, and flow out into the receiver, S. When the liquor in the tank, N, appears exhausted, it is no longer returned into the still, but is rejected as useless, and fresh quantities of wine are run in by turning the stopcocks of the pipe, P, and the distillation continued, till the accumulation of tartar and colouring matters renders it necessary to discharge the whole contents, so as to guard against the stills becoming furred.

Fig. 17. Fig. 18.



The vinasse is drawn off by turning the taps, *r r′*, and the stills washed by pumping water into them till it comes through clear. This apparatus was found to answer well; but it was not continuous in its operations, and hence much time, labour, and fuel were lost by allowing stills to cool, for the purpose of discharging the spent liquor, recharging, and again raising the temperature sufficiently to effect distillation.

Subsequently to the introduction of ADAM's and SOLIMANI's stills, improved ones were announced by BERARD and others. BERARD's still was not so complex as either of those mentioned, and it was more easily managed; but the loss in fuel was considerably greater, as the operation had to be arrested several times for the purpose of discharging and replenishing the still.

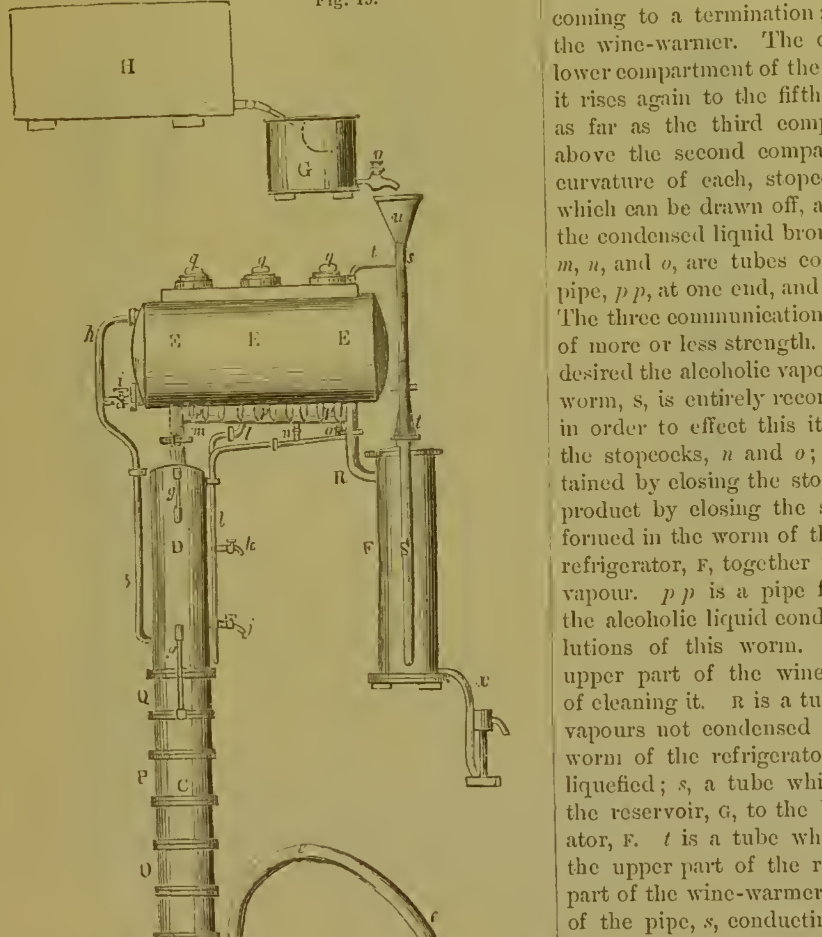
BAGLIONI was the first to conceive the idea of constructing an apparatus by which continuous distillation might be carried out. His attempt, however, was not very successful; but the subject was taken up by CELLIER BLUMENTHAL, who constructed an apparatus which was found to possess, in an eminent degree, all the requirements.

The still constructed by BLUMENTHAL afterwards became the property of DEROSNE, who so very much improved it with respect to continuous distillation, that it in a great measure superseded all the preceding distilling apparatus. The following is an account of DEROSNE's still on BLUMENTHAL's principle.

Fig. 19 is a general view of the apparatus. It is composed of seven principal parts:—The boilers, the distilling column, the rectifying column, the condenser and wine-warmer, the refrigerator, the vat where the wine is contained, and the vessel which determines the flow of wine into the apparatus. Of these, A and B are the boilers, encased in masonry or brickwork. The fire is applied under A, and the extra heat is communicated to B by the flue passing under it in its way to the chimney; C is the column of distillation; D the column of rectification; E E the condenser and wine-heating vessel; F the refrigerator; G is a vessel to furnish the wine to the refrigerator. This vessel supplies itself, by means of an automatic tap, from the store-vat, H, where the wine to be distilled is kept. The cock of this part is regulated by a floating ball, which closes it when the liquid rises in G. I is a tube of communication, conducting the alcoholic vapours from the rectifying column, D, to the worm in the condenser, or wine-heating vessel.

The stopcock, *a*, carries off the spent wine from the boiler, E. When the operation is in progress this cock is always open, and the exhausted wine flows off continually; *b* is a gauge-pipe, which indicates the height of the liquid in the boiler, A; *c*, a safety valve or pipe, to show the pressure on the boiler, A; *d*, a stop-cock, which allows the liquid from the boiler, B, to flow into the bottom of the boiler, A; *e e* is a tube that conducts the alcoholic vapours formed in the boiler, A, to the bottom of the boiler, B; the vapour, in passing through B, condenses in part, at the same time heating the liquid; *f* is a gauge to show the level of the liquid in the boiler B; *g*, *g*, level gauges, indicating the height of the liquid in the compartment of the rectifying column, D; *h*, a tube, conducting the wine from the lower part of the condenser, E, to the topmost beveled plates in the interior of the distilling column; *i* is a stopcock, by which all the heated wine in the wine-heating vessel or condenser, E, flows into the column, C, when the distillation is coming to a termination; *l l* are tubes, adjusted to the wine-warmer. The one descends as far as the lower compartment of the rectifying column, whence it rises again to the fifth; the other tube descends as far as the third compartment, and rises again above the second compartment. At the point of curvature of each, stopcocks *j* and *k* are fixed, by which can be drawn off, at will, the small portion of the condensed liquid brought back into the rectifier. *m*, *n*, and *o*, are tubes connected with the inclined pipe, *p p*, at one end, and the pipes, *l*, *l*, at the other. The three communications serve to produce a brandy of more or less strength. If a very strong spirit be desired the alcoholic vapour that is condensed in the worm, S, is entirely reconducted to the rectifier, D; in order to effect this it is only necessary to open the stopcocks, *n* and *o*; a spirit less strong is obtained by closing the stopcock, *o*, and a still weaker product by closing the stopcock, *n*; for the liquid formed in the worm of this cylinder flows off to the refrigerator, F, together with the stronger alcoholic vapour. *p p* is a pipe for receiving the whole of the alcoholic liquid condensed in each of the revolutions of this worm. *q q q* are manholes in the upper part of the wine-warmer, for the purpose of cleaning it. R is a tube conducting the alcoholic vapours not condensed in the wine-warmer to the worm of the refrigerator, F, where they are wholly liquefied; *s*, a tube which supplies the wine from the reservoir, G, to the lower part of the refrigerator, F. *t* is a tube which conducts the wine from the upper part of the refrigerator, F, to the upper part of the wine-warmer, E. *u* is the funnel-opening of the pipe, *s*, conducting the wine from G to the refrigerator; *v*, a stopcock, regulating the flow into the tube, *t*; *x*, a tube conducting the finished spirit from the refrigerator; it is so constructed that an areometer adjusted to it always indicates the strength of the brandy.

Fig. 19.



The interior arrangement of the distilling column is represented in Fig. 20. The surface of the liquid descending through this column is greatly increased by flowing in a thin stratum over the several plates successively, and the alcohol it contains eliminated with great facility by the ascending hot vapour. There are three openings at O P Q (Fig. 19) for cleaning the inside. Ten pair of copper plates are enclosed in this cylinder, and placed in such a zigzag way as to recline downwards alternately, as seen in the section; the liquid, entering at the top, falls over each of the plates in succession, thus making a longer course, and becoming more exposed to the hot vapour.

Fig. 21 is a sectional, and Fig. 22 an exterior view of the rectifying column. Six inverted vessels occupy the interior, and are so arranged that the alcoholic vapour is forced to traverse a thin layer of liquid in each. The condensed liquid returns to the column, C, and the uncondensed vapour passes onwards to the worm in the first condenser by the pipe I.

Fig. 20.

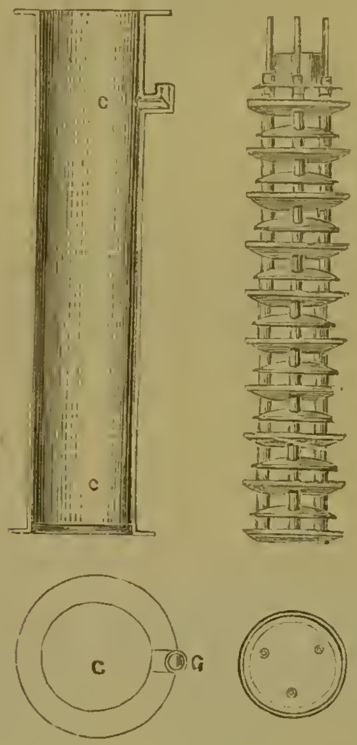


Fig. 21.

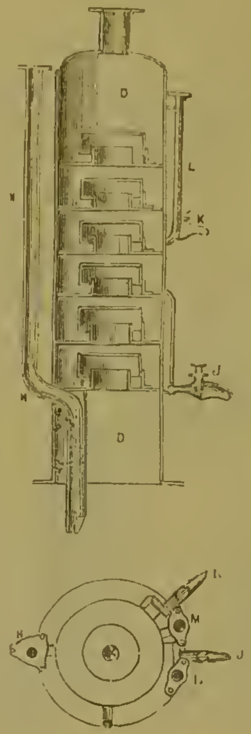
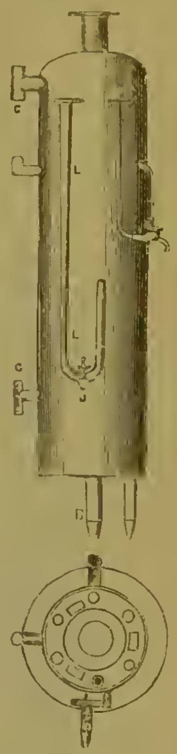


Fig. 22.



In these figures the position of the pipes, L, L, can be better seen; in the front view one is represented descending to the lower and rising again to the third vessel, and the other coming to the third and mounting again to the second; the sectional figure shows the communication of these pipes with the interior of the cylinder. G G are cases for the glass gauges; and the sketches at foot are plans showing the position of the respective tubes, here indicated by dark spots, and the letters, H, M, K, J, and L.

Fig. 23.

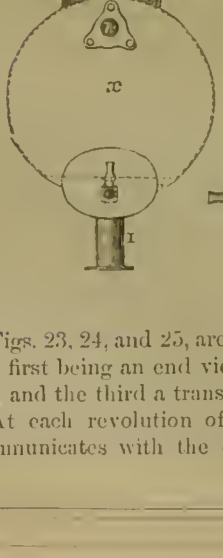


Fig. 24.

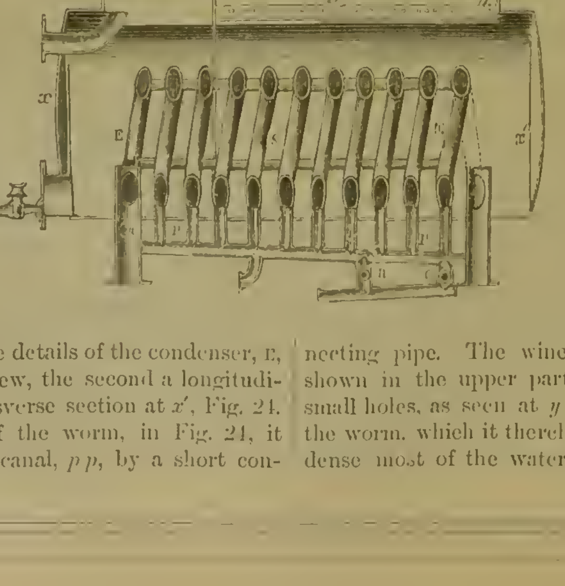
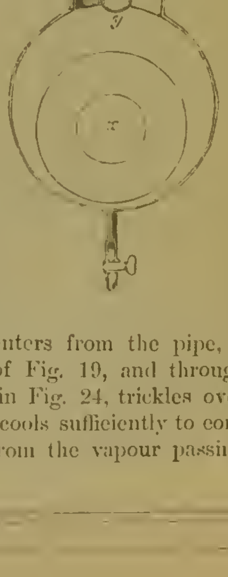


Fig. 25.

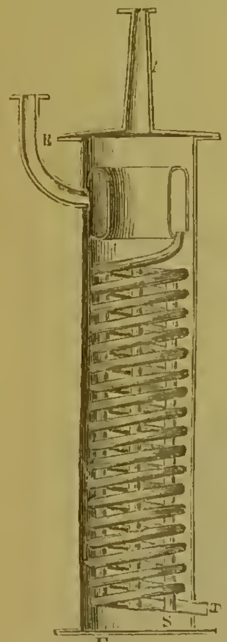


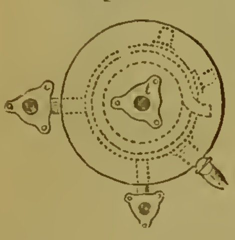
Figs. 23, 24, and 25, are details of the condenser, E, the first being an end view, the second a longitudinal, and the third a transverse section at x', Fig. 24.

At each revolution of the worm, in Fig. 24, it communicates with the canal, *p p*, by a short connecting pipe. The wine enters from the pipe, *t*, shown in the upper part of Fig. 19, and through small holes, as seen at *y y* in Fig. 24, trickles over the worm, which it thereby cools sufficiently to condense most of the water from the vapour passing through it. Fig. 26 shows the arrangement in the refrigerator, S; and the cut appended a view of the top of this column. In these figures the same letters indicate the same parts as in the general view, Fig. 19.

Referring, therefore, to Fig. 19, in connection with the parts shown in detail, when distillation is to commence the boiler, A, is filled with wine through K, to within 2 or 3 inches of the top, as indicated by the gauge, *b*; the fire is lighted, the stopcock, *v*, opened, and the wine allowed to flow into the refrigerator, F, thence into the condenser, E; when this vessel is filled, the liquid flows through the overflow pipe, *h*, into the distilling column, C, and lastly reaches the boiler, B. It is permitted to flow till it rises to within 5 or 6 inches of the top of the gauge-pipe, *f*, when the cock, *v*, is closed. As soon as the liquid in A boils, alcoholic vapour escapes by the pipe, *e*, into the bottom of the boiler, B, where the first condensation takes place, the liquor in this vessel becoming richer in spirit. In a short time the liquor in B, in consequence of heat it derives from the traversing vapour, and of its being rich in alcohol, begins to boil; part of the vapour rising through the distilling column and rectifier is condensed, while the uncondensed portion passes into the condenser through the pipe, I. When the liquid in the condenser becomes so hot that the hand cannot rest in contact with the outer case, the stopcocks, *a*, *d*, and *v*, are opened, and the wine allowed to flow till it reaches the boilers, B and A. As it descends through the distilling column, it is divested of the greater part of its alcohol by the ascending vapour; during its stay in the boiler, B, almost all the remaining quantity is removed, and the very last traces are separated in the first boiler, so that it is completely exhausted as it flows off by the waste stopcock, *a*.

Fig. 26.





In practice, however, it is found that the charge entering into the first boiler from B, if allowed to run off at the full bore of the discharge-cock attached to boiler A, would retain small quantities of alcohol. If the alcoholic liquor remained always in an upper stratum of the liquid in the boiler, then the tap, *d*, might be left open; but in consequence of its constant circulation, no such division can be expected. On this account, therefore, it is absolutely necessary to shut the discharge-cock of boiler A while the liquid is in a state of ebullition, and to slacken the fire while the spent liquor is drawn off from the boiler.

St. Marc, a veterinary surgeon attached to the personal staff of Bonaparte, contrived a still to carry out the principle of uninterrupted distillation. After the battle of Waterloo, St. Marc turned distiller in France, and about the year 1823 he removed to England. Here he and a few others formed a company for the manufacture of brandy from potatoes, and erected large works near London; the project failed, after three years' operation on a very extensive scale.

St. Marc, however, whilst the works were extant, effected many valuable improvements in the form of his still. In 1827 he obtained a patent for the United Kingdom.

This still came into great favour among the principal distillers of London, Bristol, and other towns, though for the distillation of wash it ranks far behind Coffey's. It is said that at the establishment of Messrs. Nicholson at Clerkenwell, one of these stills produced 1000 gallons of gin hourly, the cleansing and flavouring processes proceeding at the same time. It was in great demand, also, for the distillation of rum in the West Indies and several other English colonies, as by its use considerable outlay in fuel, puncheons, freight, and shipping charges were dispensed with.

Figs. 27 and 28 are sectional and front engravings of the still. It consists of seven coppers, placed one above the other, and numbered in the section, 1, 2, 3, 4, 5, 6, 7, of which six contain the wash or liquor to be distilled, and the seventh or upper one water. The coppers, which are held together by flanges and bolts, communicate with each other by the double tubes, A A, through which the vapour ascends, and also by the pipes, B B, by means of which the wash descends from one copper to another in succession, beginning with N 6, into which it is introduced by a pipe and tap, C, from the wash-charger, D. The lowest copper constitutes the body of the still, and receives the heat of the fire, and does not differ from the ordinary boilers; but the second and third coppers each contain four of the double tubes, A, and two of the pipes, B. The fourth, fifth, and sixth coppers have likewise the pipes, B, but have only one double pipe, A, in each, placed under hemispherical domes, E E E, constructed upon, and tightly flanged and bolted to the coppers that have just been mentioned. Six spiral tubes, or vertical worms, F, of which only one appears in the engraving, conduct the vapour from the upper dome through the water in the top division; these communicate with the chamber, G, capped by a small dome and pan, which is kept replenished with cold water, and the portion of vapour remaining uncondensed passes out to the common condensing worm, by the large pipe, H. I shows a pipe and stopcocks for conveying water to the top copper, No. 7, and chamber, G, and any waste water that may be used for scalding or cleaning the backs is carried off by the pipe, K, which is furnished with a branch pipe and stopcock. Water is conducted from the upper copper through the pipe and stopcock, L, to the several lower compartments into which it is introduced by means of the stopcock, M, appended to each; the water is used for bringing down the wash at the close of a back, as well as for cleaning the coppers. The manholes marked N admit of the apparatus being thoroughly cleansed, and facilitate repairs.

The first three coppers—of which the second and third only are intersected with double pipes—distil almost at the same time; the lowest, being that submitted to the action of the fire, operates on the others by the discharge of its vapours, which, ascending by means of the pipes, passes into the wash, and is there condensed, parting with its heat to the latter liquid, which is thereby quickly brought to the boiling point. The uncondensed vapour from the second compartment passes into the third with similar effect. The new vapour, necessarily more alcoholic than the first, ascends into the fourth section, where it is received under a hemispherical dome, which prevents it communicating directly with the cold wash contained in that copper. In this place the greater part of the water is condensed, yielding its latent heat to the wash which surrounds the dome, and bringing it to a higher temperature. The spirituous portion, which passes into the fifth section, experiences the same change as the vapour in the fourth dome, and so on to the uppermost, the alcoholic vapour becoming stronger as it traverses every succeeding dome. The condensed portion in each dome returns through the coppers to the third chamber, and meeting with the ascending hotter vapours, they are partially redistilled in their progress. In the third copper a second distillation commences, giving off anew its alcohol.

Fig. 27.

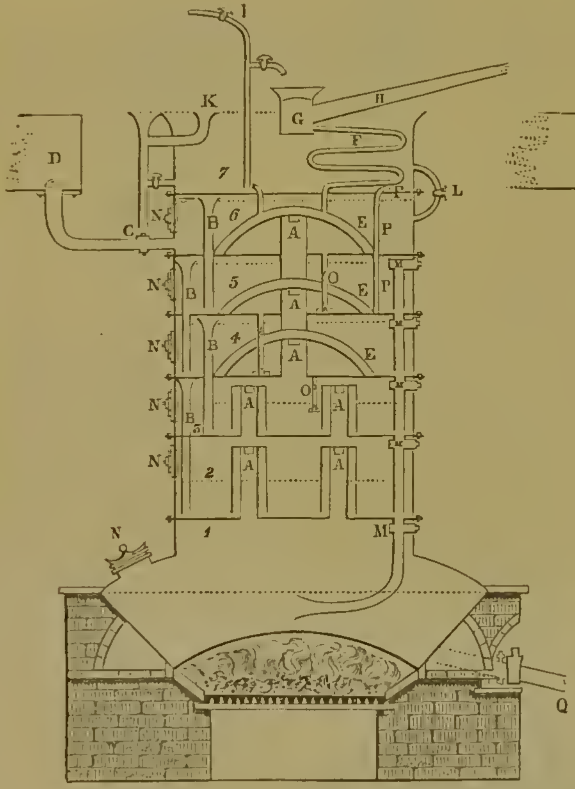
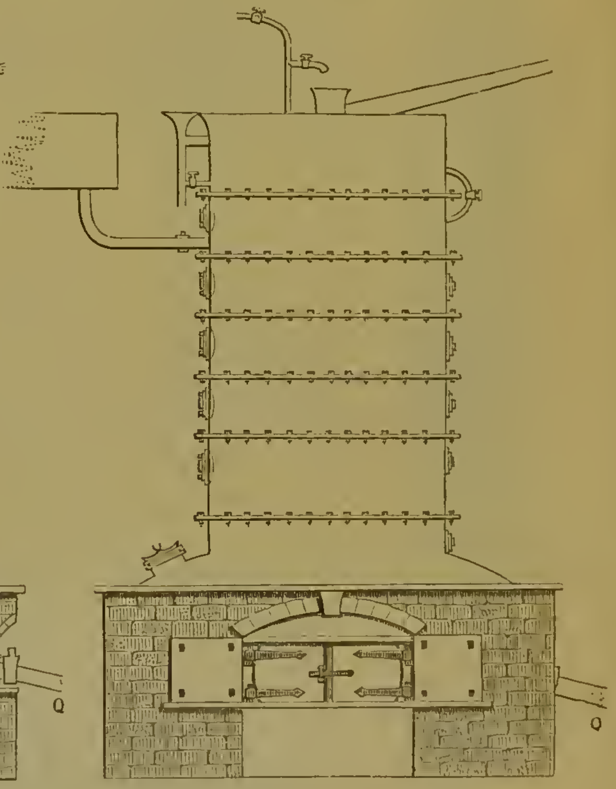


Fig. 28.



A large portion of the spirit being distilled by steam heat, is consequently more pure than that obtained by the ordinary apparatus. Therefore, all the bad flavour that arises from long and violent exposure of the wash to the action of the fire is obviated by this process. One distillation only is effected by the fire; and this is immediately succeeded by two steam distillations, and subsequently by four purifying consecutive processes, which divest the spirit of all impurities, and it comes over, at one operation, of the strength of 35 or 40 per cent. over proof, by Sikes' hydrometer.

A still of seven compartments, such as described, will not produce spirit stronger than 35 or 40 per cent. above proof; but by increasing the number of coppers or sections, a much stronger liquor might be obtained by a single operation.

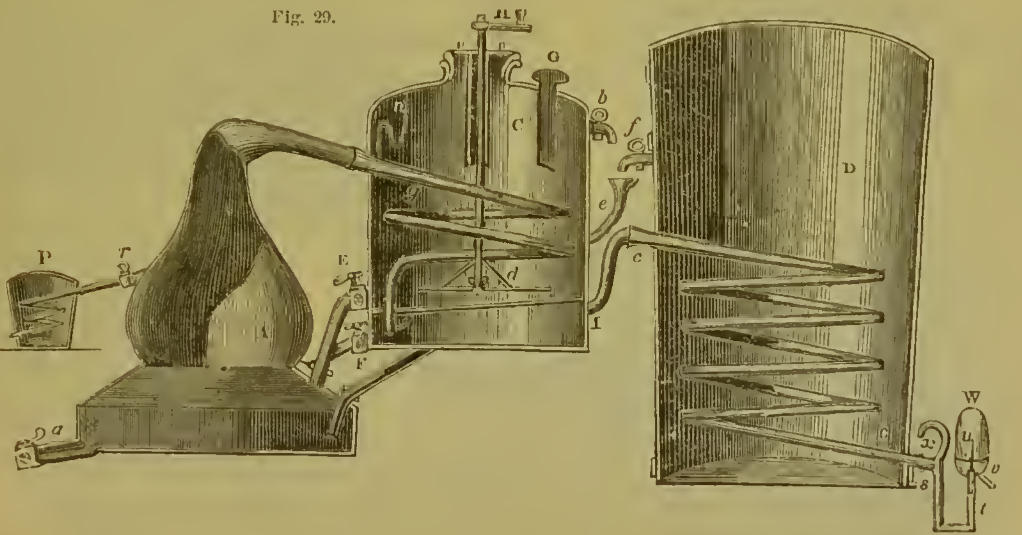
To ascertain the precise time for charging, after the exhaustion of the wash in the lower copper, the proof tap placed in its side is opened, and if the vapour issuing from it will not ignite on the application of a candle, a fresh charge is deemed to be wanting. The discharge of the spent wash from the lowest vessel, the supply from the next copper to replace it, and the opening of the tap in the pipe communicating between the charger and the top of the still to admit more wash, are all the work of about a minute, during which distillation never ceases.

The Germans still use, when working on a small scale, an apparatus for the distillation of the fermented extract of malt, which bears the name of DORN'S still, Fig. 29.

The principal parts are the body of the still, A, the wort-warming vessel, C, and the condenser, D. The still is furnished with a discharging-cock, *a*, and a small pipe with stopcock, *r*, is inserted in the head, which pipe is connected with the end of a small worm in the tub, P. C is an iron or copper vessel, furnished with a double bottom, the one 12 inches above the other. In the upper part of this vessel are contained a few coils of worm, *g g*, the lower end of which passes downwards through the first bottom to within one or two inches of the other. An opening at the top of the wort-warmer receives an agitator, H, which may be turned by the hand; a cross bar, *d*, at the end of this upright rod, stirs up any sediment which may settle on the bottom. The opening, G, serves to charge the vessel with liquor; E and F are communications between both compartments of the vessel, C, and the body of the still, for the purpose of supplying it with the liquor contained in both these parts for distillation; *b* is an overflow pipe and stopcock, by which it is ascertained when C is replenished. A pipe, I, issues from the far end of the lower part of the vessel, C, and is connected with the second worm *c*, *c*, in the large worm-tub, D. Another pipe, *e*, enters the preparer for the purpose of cleaning it with water at the termination of the distillation; the water is run into the boiler of the still, and drawn off at the discharge-cock, *a*. A pipe from an adjacent cistern, or reservoir, supplies the large tub, D, with cold water, and from this tub water is supplied to the pipe, *e*, through the stopcock, *f*.

An apparatus is furnished at the end of the worm, as it issues from the tub, in order that the flow of the liquor may be observed, and its strength noted at the same time. It consists of a tube, bent at right angles, as at *s t*, the upper part of which terminates in a curve, *x*, through which the air of the worm is expelled. The arm, *t*, is terminated in a basin, holding an inverted glass jar, W, in which a hydrometer, *u*, is placed, and floating in the spirit, in order to tell the proper strength. The pipe, *v*, carries off the finished spirit into the tank.

Fig. 29.

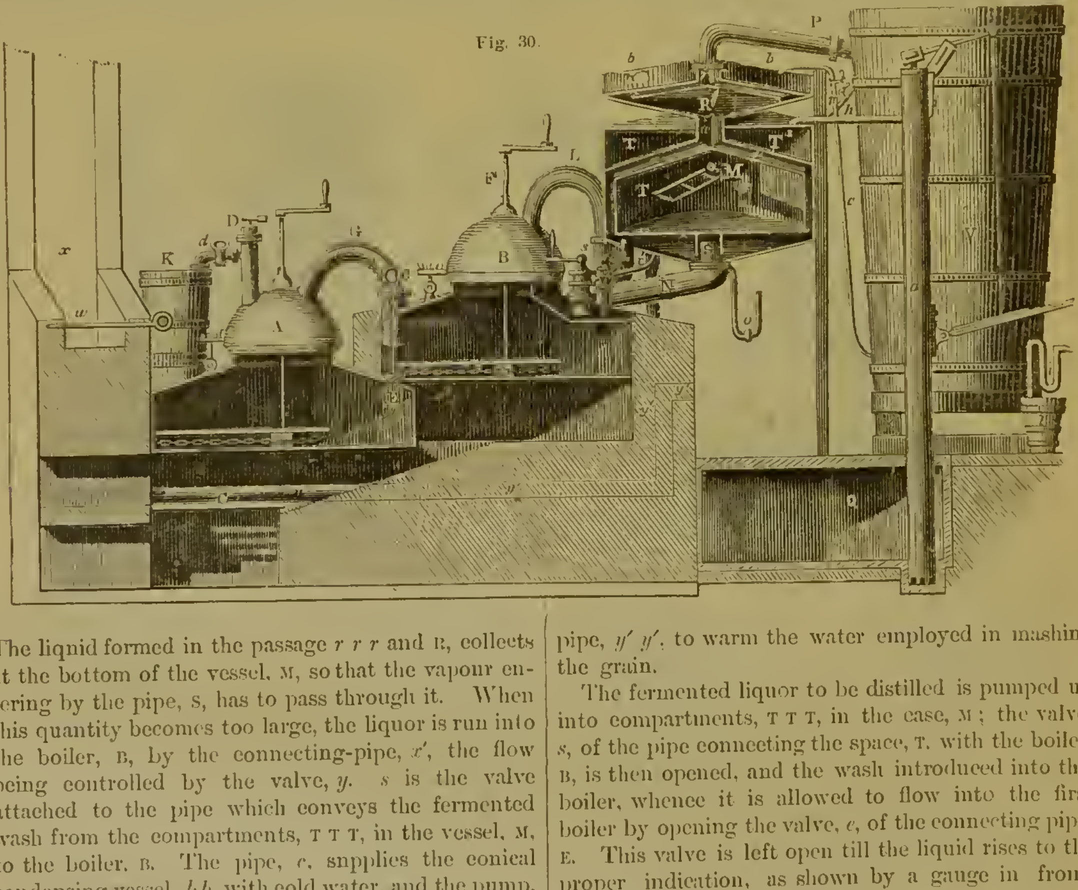


Distillation is begun by filling the wort warmer, C, with liquor, through the pipe, G, till it flows out at the stopcock, *b*, after which the cock, E, is opened till the still is filled to an overflow-pipe, which regulates the amount of liquor to be introduced, but which is not seen in the section. The cock, E, is then shut off; G is closed by a screwed cap or plug, and the furnace lighted under A. As the alcoholic vapour rises, it is partly condensed in the few coils of worm in the vessel, C, the liquid falling down to the bottom compartment; and the liquor in the vessel is heated by the latent heat of the vapour. As the liquor collects in the bottom part of the vessel, the uncondensed vapour gurgles through it, and further deprives it of aqueous vapour; the uncondensed portion then issues through the connecting-pipe, I, to the large worm in the condenser, where it becomes wholly liquefied; the excess of liquid in the lower part being returned to the still by the pipe, F. When the whole of the alcohol has been expelled, the charge in the still is emptied through the discharging-cock, *a*, a fresh supply of the heated liquor from C introduced as before, and a second operation commenced, the fire being slackened while this part of the work is going on. The small condenser attached to the boiler or still is to test whether the charge is wholly exhausted; and the small pipe, *n*, permits the escape of the air in the worm and lower part of the compartment in C.

Since the introduction of a better apparatus, DORN'S is seldom used by the large distillers: it is, however, met with in small establishments, and where brandy is rectified.

The apparatus generally employed throughout Germany for the distillation of fermented worts, &c., is that represented in section in Fig. 30, and known as the PISTORIUS still. In the figure, A and B are two boilers, connected by the pipe, G. These boilers are each furnished with an agitating chain apparatus, F, F′. C is the fire-grate; after heating the boiler, A, the flue winds under B before entering the chimney, *x*. D is a safety valve attached to the first boiler, which is furnished with a stopcocked pipe, *d*, communicating with a small worm in the condensing tub, K; by this means the distiller is enabled to ascertain when the charge is divested of the whole of its alcohol. The boiler, A, is charged from the contents of the second boiler, B, by means of a connecting-pipe, E, having a valve appended, the handle of which is seen at *e*. A large pipe, L, issues from the head of the boiler, B, and is connected with another pipe, N, of a larger calibre, having another smaller pipe, S, connected with the other end. The alcoholic vapour from the two boilers, A and B, passes through these tubes into the rectifying vessel, M, supplied with a second bottom, from which descends a vertical cylinder over the pipe, S, nearly to the exterior bottom. This passage is made of sheets of copper of nearly the same breadth as the vessel, soldered to an end plate of a few inches in breadth. The remaining parts of the vessel, noted by T T T, are charged with wash for the purpose of heating it before distillation. The spirituous vapour, on passing through this rectifying vessel, loses much water by condensation; but the chief quantity is separated in the double conical space, R, which is surmounted by a vessel of the same form, *b b*, filled with cold water from the large condensing tub. Sometimes the spirit is made to traverse two or three vessels of this kind before passing to the condenser. The pipe, P, conducts the uncondensed alcoholic vapour to the large condensing worm in the tub, V.

Fig. 30.



The liquid formed in the passage *r r r* and R, collects at the bottom of the vessel, M, so that the vapour entering by the pipe, S, has to pass through it. When this quantity becomes too large, the liquor is run into the boiler, B, by the connecting-pipe, *x′*, the flow being controlled by the valve, *y*. *s* is the valve attached to the pipe which conveys the fermented wash from the compartments, T T T, in the vessel, M, to the boiler, B. The pipe, *c*, supplies the conical condensing vessel, *b b*, with cold water, and the pump, *a*, raises the fermented wash from the tank, Q, beneath, and discharges it into the vessel, M, by the pipe, *h*. When the space in the interior of M requires cleaning, water from the condensing tun is run in through the pipe, *m*, by turning the stopcock. The siphon tube, *o*, is for the purpose of expelling the air from the apparatus when the fire is lighted under the boiler, A; it is closed by the stopcock when the alcoholic vapour reaches it.

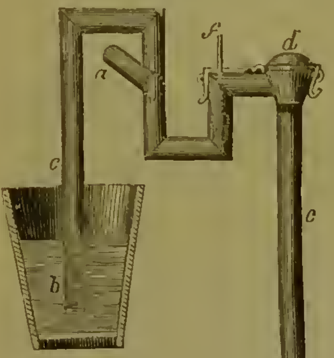
The waste heat from the furnace is made by the pipe, *y′ y′*, to warm the water employed in mashing the grain.

The fermented liquor to be distilled is pumped up into compartments, T T T, in the case, M; the valve, *s*, of the pipe connecting the space, T, with the boiler, B, is then opened, and the wash introduced into this boiler, whence it is allowed to flow into the first boiler by opening the valve, *e*, of the connecting pipe, E. This valve is left open till the liquid rises to the proper indication, as shown by a gauge in front, but not seen in the figure; all the taps are then closed. When this is done the fire is lighted. After a short interval, the liquor in A begins to boil, and the vapour passes over into B, whose contents are also raised to ebullition, which takes place at a lower degree in consequence of the quantity of alcohol it receives from it.

The alcoholic vapour from B passes over by L through the pipe, N, into the rectifying spaces, *r r r r*, where the excess of the watery vapour is condensed, and falls to the bottom of M. This liquid being the product of the former two distillations is very rich in alcohol; and when it accumulates sufficiently to cover the end of the pipe, S, the hot steam from the boilers bubbling through it disengages a very strong spirit; thus a third distillation is effected. The uncondensed portions rise and pass from the conical-shaped condenser, R, into the large condensing worm in the vessel, V, where they are completely condensed, and flow out at the lower end of the worm into the vessel, or receiver. The three distillations which here take place give a very strong spirituous liquor, and of a very good quality.

At the end of the great worm is a water-tap similar to that shown in Fig. 31. In this diagram, the end of the worm issuing from the great condensing tub is seen at *a*: *c c* is a zig-zag pipe, one end of which is immersed in a bucket of water. In the upright part of the discharging-pipe is an alcoholometer, *f*, by which the strength of the distilled product is ascertained; *d* is a watch-glass covering the funnel-opening in the larger end of the tube, which is used for the purpose of seeing the bulk of the stream of liquid which flows through the worm. By this contrivance the air is prevented entering the worm, and therefore no acetic acid is produced.

Fig. 31.



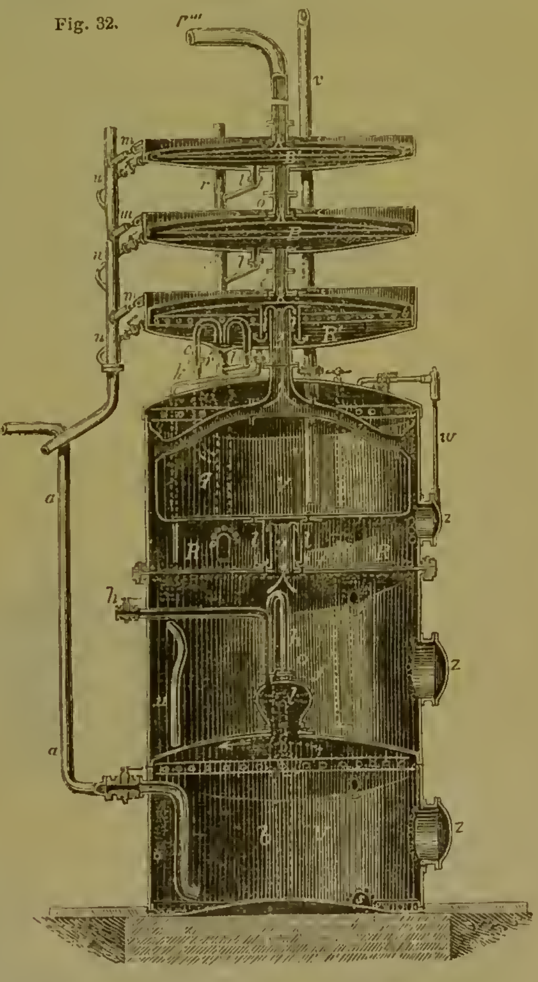
When, on turning the vapour into the small worm in the vessel, K, and collecting the condensed portion, it is found that no more alcohol is contained in the contents of this boiler, the spent liquid is drawn off by the discharge-pipe, a fresh charge admitted from the boiler, B, and this in turn refilled from the compartment in the rectifying vessel, M. During the discharging of the liquid in boiler, A, and its refilling, the fire is slackened, by inserting the damper-plate, *w*, into the flue to cut off the draught.

Fig. 32 is a modification of the Pistorius still. Though widely different in form, the principle upon which it is designed is precisely the same. U is the first still, the top of which forms the bottom of the second still, O. The top of U has in the centre a helmet-shaped headpiece, I, out of which springs the pipe *h*, which turns sharp upon itself and descends to the deepest part of the upper still, O, as shown by the dotted lines.

The vapours from the second still pass into the rectifier, R, through the wide tube, *y*, covered with the cap, *l*. From R the vapour passes through the tube, *u*, round the wort-warmer, V, and proceeds through the capped tube, *y*′, *l*, into the basin, R. In this vessel, as in the cap, *l* indicates a second rectification is effected. From R′ the vapours pass through the pipes, *o* and *o*, into two other rectifying vessels, B and B′, and proceed lastly through the pipe, *r*′′′, to the condenser.

The wort-warmer, V, is filled with worts from the mash tuns by the pipe, *v*. The glass gauge, *w*, is to indicate the height of the wash: unless its lower opening is very wide it is apt to be blocked up with the malt sediment, and to be fallacious. The wash flows from the preparer, V, through the valve, *p*, and the pipe, *f*, into the second still, O, whence it descends into the first still, U, by the pipe, *h*, shown in the engraving by dotted lines. The exhausted wash is withdrawn through S. The pipe, *h*, on the helmet, I, serves to withdraw a portion of vapour to a worm, to test the progress of the operation.

Fig. 32.



The distillation is performed by means of a current of high-pressure steam introduced into the still through the pipe, *a*. This pipe, and also the pipe *h*, is carried to the bottom of the still, and directs the steam horizontally, so that the wash is kept constantly in motion.

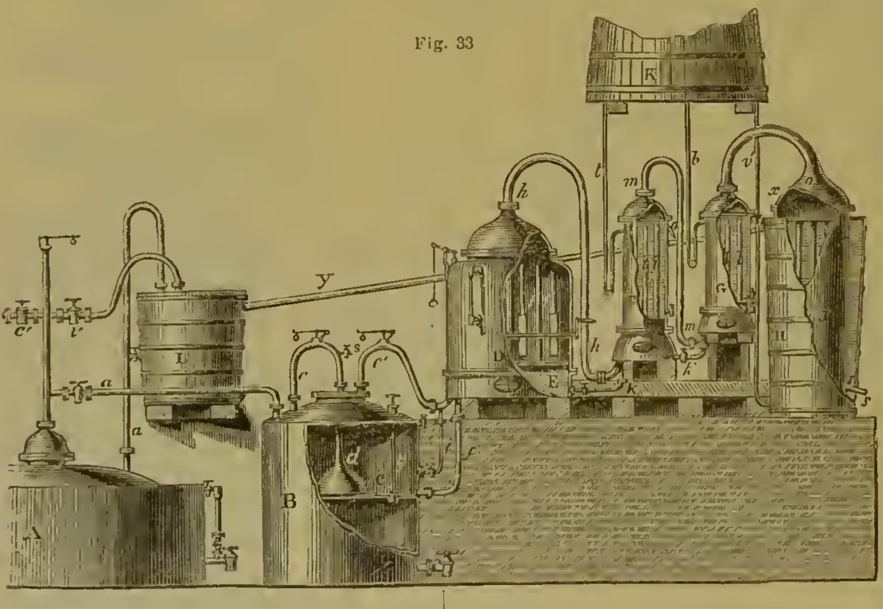
The tube, *u*, shown in the second still, O, leads the liquor from the rectificator, R, back into O, when an outside tap not seen in the engraving is opened. The liquor condensed in the basins, B and B′, flow into the discharge pipe, r'', through the pipes, *l l*; thence they are led at pleasure either into the rectifier, R, or the upper rectifier, R′. When it is desired that the upper rectifier should receive them, the tap shown in dotted lines in the engraving is closed, upon which the liquor is forced up the lowest pipe, *l*. The level of the liquid is regulated by the bent pipe, *c*, which also leads into r'', and serves to convey the overflow into R, so soon as the liquid covers the bend. This pipe also acts as a siphon, and empties the whole of the liquor into R if a rectifier. The pipe, *k*, also serves to empty the upper rectificator, R′, into the lower R, at any time when desired.

Cold water is supplied to the rectifying basins by the pipes, *n n n*, and the hot water drawn off by the pipes, *m m m*. The manholes for cleaning the apparatus are shown at *z z z*.

A form of steam distilling apparatus, which is in very general use in the south of Germany, was devised by SCHWARTZ, and is represented by Fig. 33.

A is a copper steam boiler, supplied with water from the vessel, L, by the pipe, *a′*. B and C are two stills, placed one over the other, and divided by a metal bottom. Steam from the boiler passes through the pipe, *a*, to the bottom of the still, B, heating the wash contained therein to the boiling point, and passing through the pipe, *c*, finds free vent by the bell-shaped enlargement, *d*, into the second still, C, also containing wash. When the wash in the lower still is exhausted it is drawn off through the large tap at the bottom of the vessel, and the still refilled with the heated wash from above by opening the valve, *g*. The alcohol-laden vapour from C then passes through the pipe, *e*, into the lower part of the wort-warmer, D. This part of the apparatus resembles the wort-warmer of the DORN still, and consists of two vessels separated the one from the other. The lower vessel, E, is a rectifier, and the upper the true wort-warmer. The latter contains eight tubes, *f f*, above and below. The lower ones are screwed into the plate which separates the wort-warmer from the rectifier, the apertures being above the highest level of the liquor. The vapour proceeding from the lower part of the wort-warmer through these tubes becomes dephlegmated by the surrounding cold liquor, and escapes through *h* into the rectifier of the dephlegmator, F, of which the construction is essentially the same as that of the wort-warmer, D. F contains twelve small tubes, *i i*, surrounded by water. A second rectification here takes place. G is a vessel of similar construction, in which the rectification is carried still further. The vapours from F enter through *m*, and after passing through the liquor enter the pipes, *l l*, from whence they proceed through *c* into the condensing apparatus.

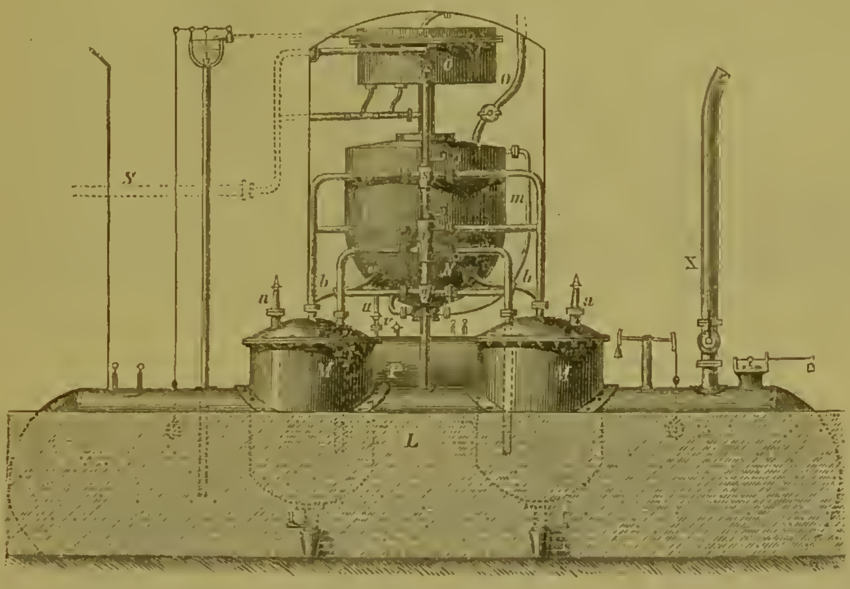
Fig. 33



The condenser, H, J, contains twelve perpendicular tubes, opening above and below upon two diaphragms. The alcoholic vapour enters from above, distributes itself through the tubes, when it is condensed, and collects in the vessel under the lower plate, whence the concentrated spirit is withdrawn by the pipe, *r*, *s*. The vessel, J, containing the tubes, is surrounded with cold water, which flows from the reservoir, K, through the tube, *r′*, to the bottom of H, whence it passes into the inner cooler, J, the tubes being thus surrounded with

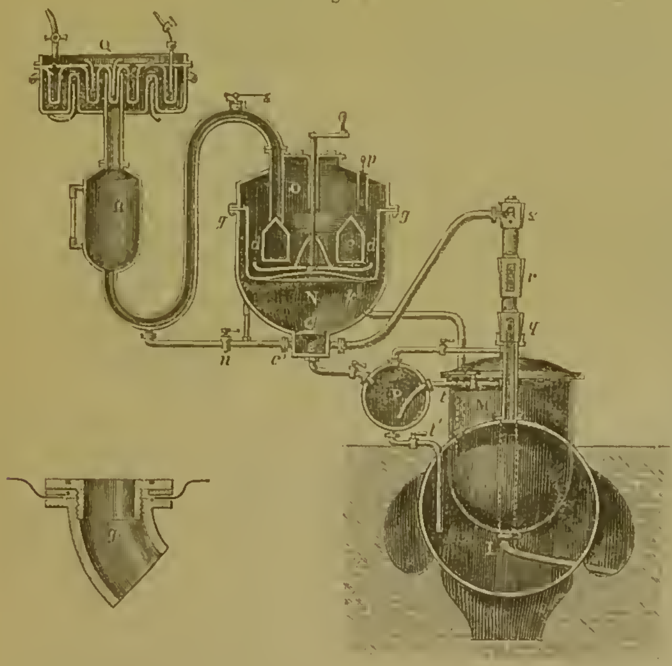
counterbalancing pressure is not maintained in the cold water. The heated water flows into the reservoir, L, through the pipe, *y*. The pipes, *b* and *t*, supply the dephlegmators, G and F, with cold water, the warm overflowing through *a* and *w* into *y*.

Fig. 34.



The opening in D, through which the wash is supplied, is not visible in the engraving. The wash, as it is warmed, falls through *f* into the upper still, and thence through *g* into the lower one. The condensed liquor in the rectifier, G, falls through K′ into the rectifier, F; the liquor from F passes through K to the rectifier, E; whilst that from E flows down through *i* into the upper still. The tap, *s*′, allows the air to escape from the apparatus whilst filling it with steam. The tap, *v*, allows of the escape of steam from the hot-water reservoir, L, or by the tap, *c*,′ it is conveyed away to be utilized in some other parts of the work. The crank, with paddles in D, is to stir the mash up, so as to keep it uniformly mixed and heated.

Fig. 35.



SIEMEN'S still, Fig. 34, is much used in Germany for the distillation of brandy. It consists of two stills, which can be used alternately. Both are bedded in the steam boiler, L. M M are the two stills; N is the rectifier of the wort-warmer; O, Q, the dephlegmator; P, a cylinder in which the condensed steam is collected for return to the boiler.

The preparer is filled with mash liquor by the pipe *o*; this again supplies the two stills by the pipes, *b b*. The refuse is withdrawn by the pipe, L (Fig. 35). From the boiler proceeds a perpendicular pipe provided with three taps, *q*, *r*, *s*, whose openings are so arranged that the steam can be directed into either still at pleasure.

The wort-warmer is shown in section in Fig. 35. It consists of an iron rectifier, or low-wine receiver, N, in which is suspended the lower part of the wort-warmer, the two vessels being bound together by bolted flanges, *g*. Steam from the tap, *s*, enters *n* through *c*, a small vessel for collecting the condensed steam, whence it is led by the pipe, *l*, into P, and thence to the boiler: *c* is covered with a finely perforated plate, to prevent sediment falling through. The vapours proceed through the tube, *e*, to the vessel, R, in which is collected the condensed liquor from the dephlegmator, Q, and then through the tube, S, to a condensing worm. The hot water from the dephlegmator is returned to the boiler by means of the tap, *w*. The tap, *t* and *t*, allows condensed water to enter the boiler.

The distillatory apparatus of Alègre is described in the Figs. 36 and 37.

Fig. 36 is an elevation of the distilling apparatus on the furnace side, and Fig. 37 a section made parallel to the visible part of Fig. 36. In these figures, A is the brickwork inclosing the fire and part of the lower boiler; B, the door of the furnace; E, the lower boiler, part of which is encased in the brickwork. F is a pipe issuing from the bottom of boiler, E, furnished with a stopcock, and serving to discharge the liquid that remains after distillation, or whenever required. *g* is a pipe and stopcock, by which the proper height of liquid to be introduced into the boiler is regulated, as shown by the dotted line, *h h*, in the section. I and C, manholes closed by a slide plate firmly fixed in its place by bolts, or by a screw-cap, for cleaning the boiler when necessary. *k* is a pipe and stopcock for ascertaining when all the alcohol has been separated from the liquid in the boiler, E.

Fig. 36.

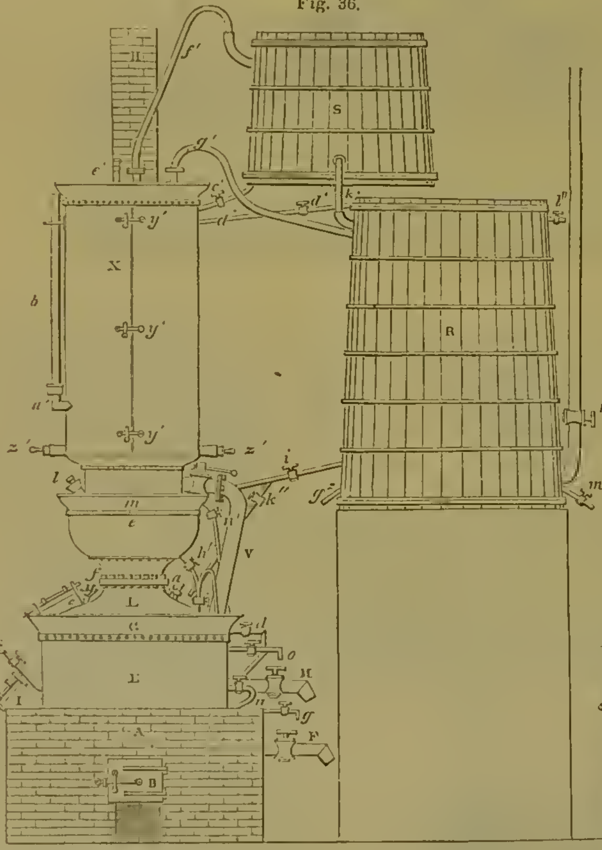
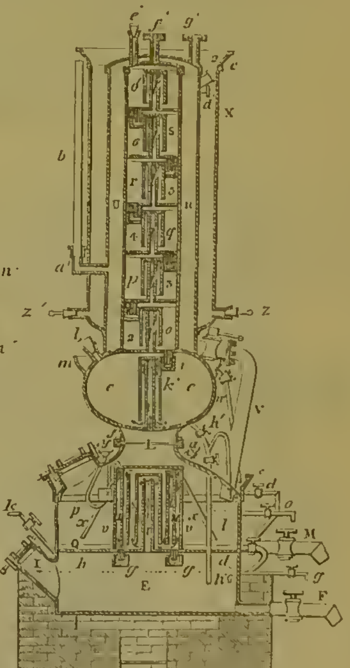


Fig. 37.



L is another boiler placed upon E, and which is emptied when requisite by the discharging-pipe and stopcock, M. Both these boilers are connected by the pipe, *n*, to which a stopcock is affixed. *o* is an overflow-pipe and stopcock, serving to regulate the height to which the liquor should rise in the boiler, L, as is shown in the sectional figure by the dotted line, *p*. Q is the stout plate or division between the boilers, E and L; in the middle of this bottom is an opening, on which is fixed the pipe, *r*; this pipe is open at both ends, and passes up through the liquid in the boiler, L, towards its neck; *s* is a hollow cylinder, which is inverted upon the pipe, *r*, and having its closed end uppermost. The open end rests on three small supports, fixed equidistant from the pipe, *r*, and elevated one inch above the bottom of the boiler, L; *t* is a third hollow cylinder issuing from the bottom of the boiler, L, and rising perpendicularly till it terminates in an open end, about one inch above the closed end of the hollow cylinder, *s*, which it surrounds; *u* is a fourth hollow cylinder, placed in exactly the same position as the cylinder, *t*, and enveloping all the foregoing; the closed end being topmost, and the lower open extremity resting upon three elevations of one inch in height, and equidistant from the cylinder, *t*. A distance of half an inch is left free between its closed end and the orifice of the cylinder, *t*. *v* is a fifth hollow cylinder, which incloses the whole of the others. Its lower extremity is fixed in the bottom, Q, of the boiler, L, and its upper extremity is attached to the closed end of the cylinder, *u*. These cylinders open alternately at the top and bottom, and by this means a passage is made for the vapour over each cylinder in succession. *x x* are tubes issuing from the top of the cylinder, *v*, at an angle of about 45°, and descending to within two inches of the bottom of the boiler, L; these tubes are open at both ends, and serve as valves to close the cylinders, *v* and *u*. Three of them are provided, which are fixed at the angular points of an equilateral triangle, supposed to be inscribed in the circular plane of the closed end of the cylinder, *v*; in the figure only two of these appear, the third being in the segment which is removed.

The safety tube, *y*, is for the purpose of preventing the influx of the liquid in the boiler, L, through the open pipes, *x x*, into the cylindrical spaces in the interior of the enveloping cylinder, *v*. Its upper extremity, which is funnel-shaped, is open to the air, the other end is in communication with the spaces in the interior of the cylinders, *v* and *u*, by passing through the closed end of the cylinder, *v*. This tube is curved so that, at or near its middle, it dips into the liquid in the boiler. In the part ascending from this curvature to the top of the cylinder, *v*, is a bulb or enlarged portion, *z*, which holds about a gallon of water introduced through the funnel-opening, *y*; *a* is a stopcock, which enables the operator to know the state of the distillation in the boiler, L, and is similar to *k* in boiler, E.

C is a circular basin placed upon the neck of the boiler, L, forming a refrigerator; *d*, a pipe and stopcock, which conveys heated water from the basin, C, into the lower boiler; *e* is a vase of an elliptical form, joined to the neck of the upper boiler by a bracket and rivets, as seen at *f*; *g g* are two tubes descending from the hollow cylindrical vessels inclosed in *v* into two small vases, or circular vessels. By these pipes the liquor condensed in the passage of the vapour over the cylinders is returned to the lower boiler, E; the small vessels act as water stopcocks, and prevent any vapour passing up by these tubes. *h′* is a tube having two stopcocks, and branching a little below the upper stopcock; by this tube the liquid condensed in the oval vase, *e*, is conducted at will into either of the boilers by opening the proper stopcock. The tube, *i*, rising vertically through the vase, *e*, to within one inch of the upper part of the vessel, is closed at the top by a plate and a hollow cylinder, *k′*, whose closed end is uppermost, and rests upon *i*. The open end of the cylinder, *k′*, terminates about one inch above the bottom of the vase, *e*. The upper part of the pipe, *i*, is pierced with a number of small holes, through which the vapour passes into the vase; *l* is a tubular opening in the elliptical vase, *e*, by which it is cleaned; this opening is closed by a wooden plug; *m* is a circular basin containing cold water, placed upon the vase, *e*, and acting as a refrigerator, and which discharges its hot water by the pipe and stopcock, *n′*.

The cylindrical column contains six compartments, or diaphragm rectifiers, *o p q r s o′*, placed one above the other; these diaphragms communicate with one another by means of six small tubes, *l′ l′ l′ l′ l′ l*, placed in their centres. The tubes are arranged similarly to the tube, *i*, in the vase, *e*, the upper part of each tube being closed, and enveloped by an inverted cylinder in the form of a hat, descending to within an inch of the bottom of each diaphragm. The *l′* tubes are perforated in the upper part, to allow the vapour to pass off, and a small pipe descends alternately at either side of the column from each compartment into a small trough or dish, 1, 2, 3, 4, 5, 6, similar to those seen at *g g*, in the lower boiler, E. By these pipes the watery liquid condensed in each compartment descends into that which is immediately beneath it, until it finally reaches the elliptical vase, *e*, and thence by the double-stopcocked pipe, *h′*, is conducted into either of the boilers at the pleasure of the operator. Hence the condensed liquor, whilst descending, offers no obstruction to the ascending alcoholic vapour. A long vertical hollow cylinder, U, envelopes the six compartments, *o p q r s o′*, at a distance of 6 inches. Water is introduced into this space, and thus the cylinder, U, acts as a refrigerator to the interior column.

The hot water is discharged from the refrigerator into the upper boiler by the large pipe and stopcock, V. X is a cylinder resting upon the projecting edge attached to the cylinder, U, which serves as its base; it is made to open and shut by the latches, *y′ y′ y′*. The space between this envelope and the cylinder, U, is used to torrefy the malt previous to grinding. The cover of the envelope is perforated with small holes, to give passage to the vapour evolved from the heated grain. *z′ z′* are two apertures by which the grain is withdrawn when properly dried. *a′* is a tube communicating with the interior of the refrigerating cylinder, U, bent upwards at right angles; at a short distance above this angle the tube is enlarged, forming a receptacle for the glass tube, *b*, serving as a gauge to show the height of water in the refrigerator. *c′*, a tube with stopcock, by which the fermented liquid is conveyed into the refrigerator when it is desired to carry on the distillation with the assistance of the cylinder, U, in conjunction with the boilers. Whether the liquid to be distilled be wine or wash, it is invariably heated here, so that it may be afterwards let down into the boilers at nearly a boiling temperature, by the large tube and stopcock, V, and the smaller one, *h′*. *d′* is a pipe and stopcock, by which wine may be introduced in the same way as the wash from grain by the pipe, *c′*, when that liquid has been employed in the course of operation. *e′* is a tube by which water is allowed to enter the rectifier for the purpose of cleaning it. *f′* is a tube, which conducts the rectified spirituous vapour from the summit of the column to the condensing worm in the superior condenser, S. *g′* is a small tube, conducting the vapour arising from the liquid in the refrigerator, U, into a small worm placed in the large condensing tun, R, collaterally with the large one. The chimney has a damper to regulate the draught from the furnace, B, which must be diminished during the charging of the apparatus.

When the apparatus is prepared, as seen in the figure, all the stopcocks are shut, excepting the overflow-pipes, *o* and *g*, which are left open.

The commencement of the operation is the filling of the large condensing vessel, R, with water, after which the superior large tub, S, is filled with wash liquor, or wine, as the case may be. This tub contains a condensing worm, wherein the vapour arising through *f′* is partly condensed, and which is connected to the large worm in the principal condenser, R, as seen in Fig. 36, by the pipe, *k′*. After S has been replenished with liquor, the lower boiler, E, is filled with water through the tubular opening, I, then the fire is lighted and the distillation of the water proceeded with, till the liquor in the vessel, S, acquires a temperature of about 100° Fahr. (37°·7 C.). As soon as the steam from the boiling water ascends to the upper boiler, the stopcock of the tube, *c′*, connecting the vessel, S, and the refrigerating space inside the cylinder, U, is opened, and the liquid allowed to flow in until the remaining liquor in the vessel also reaches 100° Fahr. (37°·7 C.). When this happens, the cock of *c′* is closed, and the vessel, S, replenished with more of the liquid. Both the stopcocks of the pipe, *h′*, are next opened, in order that the water condensed in the diaphragms of the rectifying column, as well as in the elliptical vase, *e*, may pass into the boilers; the stopcocks, *i*, *k″*, are at this time likewise opened to fill the refrigerating circular basins, C and *m*, attached to the upper boiler and vase, *e*. When these are full the cocks are closed, and the fire slackened by throwing on some moist small coal or wood, and inserting the damper. Water is first distilled in order to wash the interior of the apparatus, and to heat the liquor in the cylinder, U, and the vessel, S. The stopcocks of the pipes, F, *g*, *n*, are opened in order that the water contained in both boilers, as well as in the elliptical vase, may flow out by F. During the escape of the water the plate closing the tubular pipe, I, is withdrawn, and a broom or mop introduced to clean the bottom of the boiler. When the apparatus is newly erected this distillation of water is necessary to purify the interior from rosin and other matters proceeding from the soldering, &c.; but in other cases it is not requisite, except when impurities collect in the boilers or rectifying diaphragms, or when the distillation has been suspended for some days, or after repairs. It is customary to fill the whole apparatus with water when not required for use, in order to prevent the formation of acid, which would act on the various boilers and other parts of the whole still; the water is drawn off when operations are on the point of recommencing.

The boilers being empty, the stopcocks F and *n* are closed, and the lower boiler filled with water, until it flows out by the pipe, *g*, which is at this time closed, as well as the opening, I, and the stopcocks in tube, *h′*. The fire is then hastened by withdrawing the damper; and during the time the liquid in the lower boiler is rising to 212° Fahr. (100° C.), the stopcocks of the pipe, V, and of the overflow pipe, *o*, are opened, and the heated alcoholic liquid in the refrigerating cylinder, U, admitted, until the boiler, L, is filled, which is indicated by the liquor flowing out through *o*. These tubes are then closed, and the tube, *c′*, opened to refill the cylinder, U, with the spirituous liquor from the vessel, S, to the proper height, as indicated by the glass gauge pipe, *b′*, after which the cock of the pipe, *c′*, is shut. It is specially necessary that the water in the large condensing vessel, R, should be thoroughly cold. This is effected by opening the stopcocks, *l″* and *n″*. By turning the stopcock, *n″*, the cold water from the reservoir, which should be erected at an elevation, and convenient to the place for the use of the apparatus, flows into the lower part of R, and the hot water is discharged through the pipe, *l″*, at the same time by the influx of cold water beneath. Matters being thus in readiness, the vessels are charged, and while doing so the fire is briskly urged; the water of the lower boiler soon reaches ebullition, and the liberated steam, coming in contact with the bottom, Q, of the upper boiler, heats the spirituous liquor which it contains. As the steam is generated in greater abundance, it rises through the cylindrical pipe, *r*, and thence descends and ascends alternately in its course over the other successive cylinders, till it escapes by the open ends of the three diverging pipes, *x x x*, immersed in the liquid contained in the upper boiler. During this complicated course of the steam the contents of the boiler, L, are raised to ebullition. By this means alcoholic vapour is evolved through the pipe, *i*, and after traversing the elliptical vase, the uncondensed portions ascend into the six diaphragms, *o p q r s o′*, in the rectifying column, by means of the communications, *l′ l′ l′ l′ l′ l′*. Here the rectification is principally carried on; some of the aqueous portion of the vapour is condensed in each compartment till the spirit reaches the top of the column, from which it is carried off by the pipe, *f′*, into the coiled portion of the large worm, deposited in the vessel, S, where it is perfectly condensed in its course through this and the other worm in the large condenser, R, and flows in a fine stream into the appropriate backs, or spirit vats, placed below the protruding end of the worm at *m′*. The weak spirit from each compartment of the steam rectifier returns by the small pipes, 6, 5, 4, 3, 2, 1, till it descends into the elliptical vase, *e*, and thence through the pipe, *h′*, into the boiler, L. As the distillation advances larger quantities of vapour rise from the heated liquor, both in the boiler and oval vessel, *e*, as well as in the lower compartments of the rectifying column, the condensed water from it always descending, while the distillation of the alcoholic liquor continues to afford alcoholic vapour. When on opening the test-cock, *a*, and applying a light to the vapour, it inflames, there is still some alcohol in the liquid in the boiler, but if it does not ignite, the whole of the spirit has been eliminated; the firing is then finished, and another charge begun. The distillation of the first charge requires a period of three hours, on account of the boiler being filled with cold water; but in each succeeding operation only two hours are required, as all the parts of the apparatus, with the water in the lower boiler, are hot.

After the whole of the alcohol has been expelled, the boiler, L, is emptied and thoroughly cleansed. The opening, *c*, and the stopcock, M, are then shut, and the cock, V, opened, in order to allow the heated liquor from the refrigerating compartment in U, to flow in to refill the boiler. When the liquid has risen to the level of the pipe, *o*, the cock, V, is shut, and the communication between the refrigerating cylinder and the vessel, S, containing the liquid to be distilled, is opened by turning the stopcock, *c′*, and the cylinder refilled to the proper height, as indicated by the gauge pipe, *b′*. The connecting-pipe between the vase, *e*, and the under boiler, is opened by turning the cock, *h′*, and closing the lower one with which this pipe is supplied, in order that the liquor collected in the vase during the distillation of the preceding charge may flow into the lower boiler, whose overflow pipe, *g*, should be opened to indicate when it is full. If the quantity be not sufficient to fill the lower boiler, liquor is allowed to flow in from the refrigerating basin, C, attached to the upper boiler, by opening the stopcock, *d*. As soon as the liquid flows out through the pipe, *g*, the stopcock of this, as well as the cock of the pipe, *d*, is closed, and the fire stirred. Shortly afterwards, when the steam from the lower boiler rises through the apparatus, the stopcock of the pipe, *n′*, is opened, that the hot water contained in the refrigerator, *m*, may descend to that attached to the boiler, L. After the whole of the water is run down, the tap, *n′*, is shut, and *i* and *k″* opened, to replenish both the refrigerating basins with cold water from the large condensing vessel, R. The second firing is about this time in progress, the liquid in the boiler is deprived of its alcohol in the manner as before explained, and the distillation of the charge is completed in about two hours. A similar mode of operation to that described takes place at each charge, irrespective of the alcoholic liquor submitted to distillation.

Miller, of Glasgow, patented a still, the novelty of which lies in the employment of evaporating cones, having open spiral channels winding round their exterior. In other respects the still embodies the main features of all the principal distilling arrangements already described—namely, that of returning the products of the first condensation, which contain an excess of water, to the body of the apparatus for further rectification.

Fig. 38 shows the principal parts of the still and worm; Fig. 39, a sectional view of the cone. A is the body of the still or boiler, which is of ordinary construction. The head of the still, A′ A′ A′, consists of three cones, placed concentrically, B, C, D, but a small distance apart. The surfaces of the cones, B and D, are plain, but round the exterior of the cone, C, an open spiral channel, X, winds from the top to the base. The position which this part occupies may be better understood from the detached view of the cones in Fig. 39, where X X shows the channel, and C the cone. A pipe, E, leads from the annular space between C and D to the wash-heater, F, which consists of a vessel filled with a number of small parallel pipes, and communicating with a low-wine condenser, G, which is placed in the upper part of the worm tub. The wash is supplied to the vessel, F, by a wash-charger reaching from the backs, and communicating with the vessel, F, at M; this, however, is not shown in the figure. I is a pipe which leads from the top of the outer cone, B, to the spirit condenser, K, which consists of a cylindrical case, inclined towards the still, and containing a number of pipes laid longitudinally, through which the gaseous products pass; it is filled with water supplied by a pipe not seen in the figure; the heated water is discharged through the pipe, Q. The pipe, K′, issues from the end of the spirit condenser, and enters the refrigerator, where it is united to the worm, L, placed in the bottom of the tub, W. O and *i* connect the condensing pipes in the vessels, K, F, and low-wine condensing worm, G, with the outer part of the cone, C; and the pipe, H, serves as a communication between the top of the wash-heater, F, and the body of the still, for the purpose of charging the latter.

The following is the mode of working:—A quantity of wash is run into the still, A, from the wash-heater, F, through the pipe, H, to the height of say 3 or 4 inches. As the wash boils, the vapour arising from it ascends the space in the still head, A′A′A′, between the cones, C and D, and through the pipe, E, into the wash-heater, F; part of the vapour is here condensed, at the same time heating the liquor contained in the vessel, and the remainder passes off into the low-wine condenser, G. The condensed liquor in F, as also that which is formed in G, returns through the pipes, *i*, J, into the top of the space, B and C, in the still head, and flows into the channel, X, where it is reheated in its descent through this compartment to the base of the cone, by the simultaneous ascending vapour between C and D, and the portion thus evaporated flows off through the pipe, I, to the spirit condenser, K. That portion of the liquid which is not converted into vapour is ejected from the cone, and received into the boiler through a pipe, P, to undergo another distillation.

In the spirit condenser, K, a further rectification takes place; all the finer parts of the spirit pass off through the pipe, K′, into the worm, L, where they are condensed and afterwards discharged through R into the spirit-back, while the coarser products return through the pipe O, into the canal, X, for further purification. Whatever be the quantity of wash which is introduced at first into the still, no more should be allowed to enter till the whole of its alcohol is expelled. For this end the supply pipe, H, is furnished with a stopcock, N, and a branching pipe, S, for the purpose of drawing off any excess of wash into a tank appropriated to that purpose. Towards the end of the distillation, the weak faints may be run off from J, by means of a branch pipe, V, that enters the large condensing worm.

At Bushmills, in Ireland, a noted whisky is made exclusively from malt, which is prepared in the ordinary way, excepting that peat is used in drying it. The quantity of malt wetted for each brewing is 80 bushels, from which only one mash is prepared for fermentation, the after-washings being retained, as usual, for exhausting fresh quantities of malt. In preparing the first mash, from 18 to 20 gallons of water are used to each bushel of malt. Only as much water or small worts is run on as will wet throughout the whole of the malt, the temperature being 146° Fahr. (63°·3 C.); in about fifteen minutes afterwards the remaining quantity is run on at a heat of 155° to 160° Fahr. (68°·3 to 71°·1 C.).

After drawing off the first mash, 900 gallons of water are let down into the grist at a temperature of 170° to 175° Fahr. (76°·6 to 79°·4 C.), and after mashing for three-quarters to one hour and a quarter, the wort is let into the under-back, and pumped into the coppers: 900 gallons more are used in the third mash, the temperature being 180° Fahr. (82°·2 C.); both these liquids are used in making the first mash on the next day's brewing, and on account of the density of the small worts, the malt used is from 65 to 70 bushels.

The density of the first mash, when let into the fermenting tun, is 50 lbs. to the barrel as usual. None but the best of barm is employed in the fermentation; the quantity is 1 per cent. of the wort, one-half of which is added at the commencement, and the other when attenuation has reached 30 to 35. Attenuation is usually completed in forty-eight hours, though in variable weather a longer time is required; the quality of the malt also affects the quickness of the decomposition, but the chief cause of a good or bad fermentation is the yeast; the fermented worts are reduced in gravity to that of water, and frequently below this. The mash stills at the Bushmills factory are of the old description, and the manager states they are the best for making fine spirit, an assertion with which many will coincide.

Fig. 38.

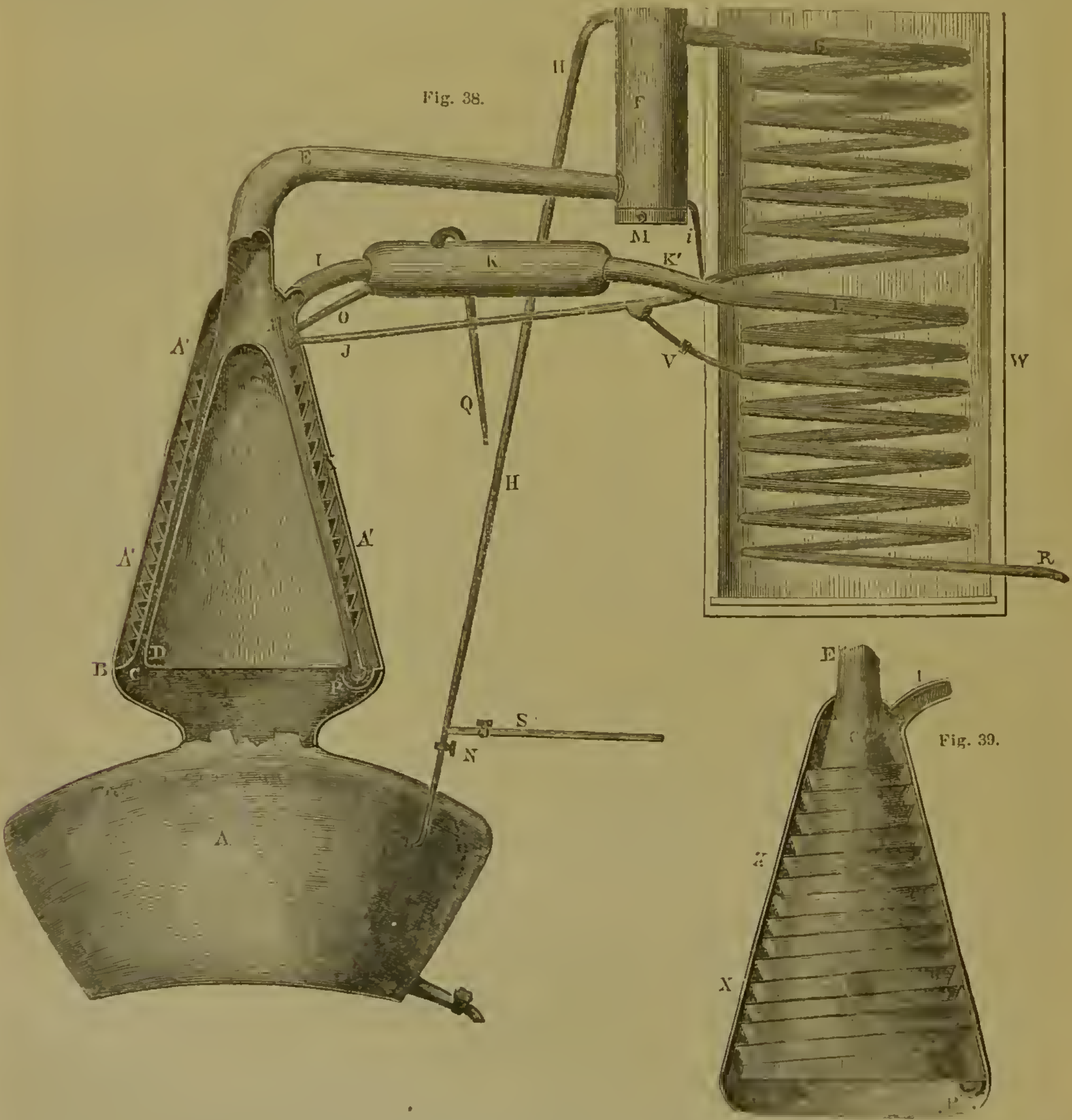


Fig. 39.

The average yield in this establishment is from 14 to 16 gallons per quarter of 8 bushels, but there is always a variation above or under these figures, according to the quality of the grist. The best spirit is made always in dry weather.

Whisky Distillery.—An Irish whisky distillery of the most modern construction is shown in "Alcohol," Plate I. This distillery has been erected at Banagher, King's County, Ireland, for the Banagher Distillery Company (Limited), under the auspices of Sir Sills John Gibbons, Bart., the chairman; Mr. J. T. Crosthwait, the resident engineer; and Mr. W. Peacey, the manager and distiller. It is probably the largest whisky distillery that has been as yet erected, and is capable of producing over half a million gallons of whisky per annum.

The engraving represents a longitudinal section, in which most though not the whole of the utensils are shown. By the aid of the following description by F. Pontifex (Henry Pontifex & Sons, who constructed the machinery), it will easily be understood:—

"The first part of the operation of whisky distilling is very similar to that of brewing beer, and the reader is referred to the article on Beer, in which will be found detailed descriptions of all the utensils and machinery common to the two processes. It may be mentioned that the 'pot still' system here described is used only with the best grain: it produces a spirit which retains the flavour of the grain throughout, and does not require any addition of essences. Coffey's patent still (which see) is used with inferior grain, and produces at one operation a strong neutral spirit, which is, however, generally deficient in character and flavour.

"As described in the early part of this article, whisky is made partly from malt and partly from unmalted grain, and the excise laws compel the two to be ground apart. The grain stores are in the engraving on the right. The grain is first brought in at the hopper near the ground. It is raised from floor to floor by the elevator, which runs up through the middle of the building, completely to the top, so that it can deliver the grain to either of the floors. The unmalted grain is stored on the top floor, and when required for use is conducted by the horizontal 'corn conveyer,' which runs in through the upper part of the distillery from the corn stores to the millstones, of which there are six pairs (three pairs only are shown) close to the still-house. The 'corn conveyer' is an endless band of india-rubber or webbing, running on rollers, but not having such buckets as the elevator has.

"The millstones are precisely similar to those used for grinding corn into flour, and in this respect the present operation differs from that in beer brewing, as in the latter the malt is only crushed between smooth rollers, and not ground. When the grain is ground, it is raised by an elevator (not shown) to the floor above the mill stones, and thence conveyed in sacks, as required, to the hopper or 'grist case' attached to a Steel's mashing machine (for description, see Beer) over the mash tun.

"From each floor of the corn-store on which malt is kept, there is communication by a hopper and shoot to a malt screw, running horizontally near the ground from the corn store to another elevator, by means of which the malt is raised to near the top of the building, and thence falls to the King's grain measurer, which gauges the quantity (see Beer), and into the hopper over those millstones which are specially used for malt. There are two pairs of these stones. The ground malt is again raised by another elevator, running by the side of the one last mentioned, and delivered to either of the two floors shown, from which it is conveyed to the Steel's mashing machine in the same manner as the unmalted grain.

"Having now brought the grist to the point ready for mashing, the provision for the water supply must be referred to. In the Banagher distillery there are five sets of three-throw pumps (a side view of one set being shown near the mash tun). Two sets of these pumps are used for water, one set for wash, one for faints, and one for spirits. The water is pumped from a well into the large iron tank that forms the roof of the still house, an opening being left in the middle with a lantern roof for light and ventilation. This tank holds about 70,000 gallons.

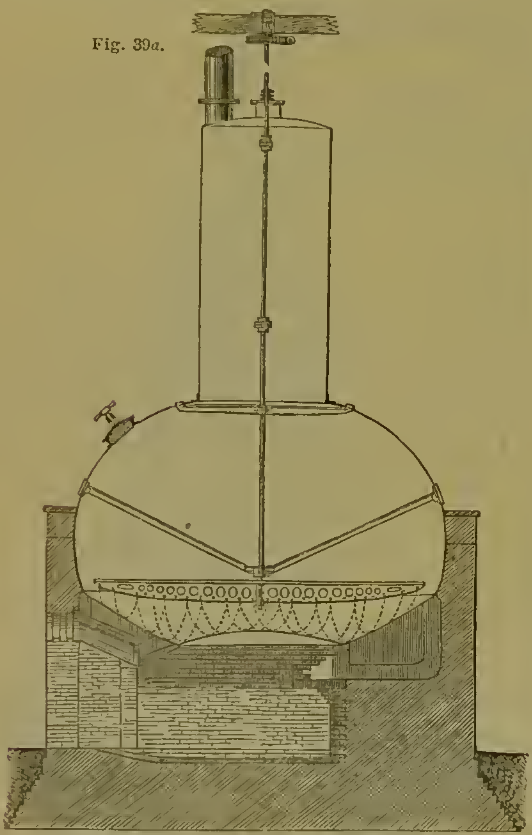
"From this huge vessel the water runs to three boiling tubs, two of which are shown on the extreme left of the engraving. Each holds about 35,000 gallons. Below the boiling tubs are shown four steam boilers, each of about forty horse-power, which are heated by Jukes' revolving furnaces. These boilers provide the steam for driving the steam engine (shown near the boilers) and for heating the water for mashing. Each boiling tub has in it a perforated copper globe connected with the steam boiler, into which the high pressure steam is brought and injected direct into the water through the perforations. The hot water, when at the right temperature, is conducted from the boiling tubs to the mashing machine and mash tun. The ground corn or 'grist' and the hot water pass together through the former and fall into the latter.

"The mash tun (there are two of them) is made of cast iron, 30 feet in diameter and 8 feet deep, and holds more than 35,000 gallons. It is fitted with a false bottom, and an internal mashing machine similar to those used in breweries. After mashing, the extract or 'wort' runs into the under back (a cast-iron vessel holding about 10,000 gallons, not shown in the engraving), and is pumped into the wash backs, seven in number, containing about 36,000 gallons each. The wash backs are simply wooden tubs similar to the boiling tubs. Here the proper proportion of yeast is added, and fermentation is continued until the wash is ready for distillation. So far the operation is much like the brewing of beer, except that the wort is not boiled. All the appliances hitherto mentioned are more fully described in the article Beer.

"Distilling proper is conducted as follows:—The wash having been perfectly fermented is run into the wash charger (a cast-iron tank not shown on the engraving), from which it is pumped up into the intermediate wash charger or heater, the cast-iron tank above and just to the left of the stills. The arm pipe (that is, the pipe leading from the still head to the worm) of the wash still passes through the intermediate wash charger, taking one turn inside it, so that the hot vapour from the still on its way to the condensing worm communicates some heat to the wash that is to be used for the next distillation. From the intermediate wash charger the fermented and partially heated wash runs into the wash still, which is made of copper, and contains about 20,000 gallons. This still is furnished with a revolving furnace similar to those applied to the steam boilers; and it may here be mentioned that the other two stills have similar furnaces.

"Fig. 39*a* is an illustration of the wash still, showing the 'rousing' apparatus inside. If not agitated, the thick particles of the wash would settle on the bottom of the still, and cause it to burn; therefore, while distillation is going on the 'rouser' is revolved, and the chains attached to it sweep the bottom of the still, and keep its contents in motion. The still head is a large copper cylinder, and from the top of it comes the copper arm pipe, which passes through the intermediate wash charger, and continues to the condensing worm, which is a coil of copper pipe immersed in cold water in the worm tub (this is the third vessel from the left of the engraving, next to and larger than the two boiling tubs shown). The vapour arising from the still is condensed in this worm, and runs out into a copper vessel called the 'safe.'

Fig. 39*a*.



"This first product is called 'low wines,' and is pumped into wooden vessels (not shown) called 'low-wines receivers;' from there it passes to the second, or 'number one low-wines still.' It is there distilled again, and again condensed as before described, and this second product is the 'faints.'

"The 'faints' are pumped into wooden vessels called 'faints receivers,' from which they are run into the third or 'number two low-wines stills' to undergo a third distillation; the product of this still being 'Irish whisky,' which is pumped into vats, and matured until ready for market.

"The machinery is worked by a steam engine, and by a turbine (not shown in the engraving), and the whole arrangements are such that hand labour is almost entirely dispensed with. The pipe connections between the various utensils, as well as the shafting, &c., for driving the machinery, are very considerable in a distillery of this size, but cannot be shown in the engraving."

In all modes of conducting distillation, it is important to bear in mind that the fermented mash is a mixture of volatile and non-volatile substances, and that the object in view is to separate one of the volatile substances only (the alcohol) from all the other bodies present. The non-volatile matters are the malt, husks, and fibre, various inorganic salts, undecomposed yeast, lactic acid, succinic acid, glycerine, &c. The volatile are mainly alcohol, water, acetic acid, and fusel oil. The difficulties encountered in separating the water have been fully described, and likewise the means now adopted for overcoming them; but in endeavouring to obtain the largest possible yield of spirit, the distiller is apt to forget that he is doubling his labour by at the same time promoting the formation of that mixture of amylic, butylic, and propylic acids, which is commonly known as "fusel oil."

Ethylic alcohol, when contaminated with fusel oil in any considerable quantity, has a very unpleasant, nauseous, and fiery flavour, and besides acts upon the human system in a very injurious manner. The quantity present appears to depend entirely on the temperature at which the last part of the distillation has been conducted, since all the constituents of fusel oil boil at a much higher temperature than ethylic alcohol. Hence, if a pure spirit is desired, it is essential to avoid pressing the process too closely as it approaches its termination.

This is the more important as the complete removal of fusel oil is a matter of considerable difficulty, and indeed is seldom effected on the large scale. Repeated distillation fails to separate the ethylic from the amylic alcohol, though the former boils at 79° C. and the latter at 132° C. It is necessary to dilute the contaminated alcohol largely with water, and to collect only the strong spirit which first comes over, and to repeat the operation several times before even a tolerably pure spirit can be obtained. The expense and trouble of this operation is, of course, very great.

The amount of alcohol to be procured by the operations of fermentation and distillation depend not only on quantity of alcohol-forming constituents, that is, the starch, dextrose, or cane sugar, of the raw material employed, but also on the proper carrying out of the important operations of distillation, mashing, and fermenting, in properly constructed apparatus, and with due judgment and care.

Potteen Whisky.—This far-famed spirit was some time ago more extensively manufactured than at present. It seems to be more than ever prized on account of its scarcity, and bears a high price. The singular flavour of the Irish spirit is supposed to be caused by using turf to dry the malt. DONOVAN describes a potteen distillery he inspected in the south of Ireland. It was a place renowned for producing good whisky. The distillery was a small thatched cabin; there was a large turf fire kindled at one end, and confined by a semicircle of large stones, upon which a 40-gallon tin vessel, serving the two-fold purpose of a water-heater and a still body, was resting. An orifice in the roof, immediately over the fire, served as a chimney for the escape of the smoke after it had traversed the apartment.

The mash tun was a cask hooped with wood, at the under part of which, next the chimb, was an opening plugged with tow. This vessel had no false bottom; in place of it young heath was strewn, and over this a stratum of oat-husks. Here the mash of hot water and ground malt was occasionally mixed up for two hours; after which time the vent at the bottom was opened, and the worts were allowed to filter through the layers of oat-husks and heath. Mashing with hot water on the same grains was then repeated, and the worts were again withdrawn. The two worts being mixed in another cask, some yeast was added, and the fermentation allowed to proceed until it fell spontaneously, which happened in about three days. It was now ready for distillation, and was transferred into the tin boiler, which was capable of distilling a charge of 40 gallons. A piece of soap, weighing about 2 ounces, was then thrown in to prevent its running foul; and the head, a large tin pot with a tube in its side, was inverted upon the rim of the body, and luted with a paste made of oatmeal and water. The lateral tube was then luted into the worm, which was of copper of 1½ inch bore, coiled in a barrel for a flake-stand. The tail of the worm, where it emerged from the barrel, was caulked with tow. The wash speedily came to the boil, and then water was thrown on the fire; for at this period is the chief danger of boiling over. The spirit almost immediately distilled; it was perfectly clear; and by its bead, this first running was inferred to be proof. Its flavour was really excellent; and it might well have passed for a spirit three months old. As soon as the upper stratum of water in the flake-stand became warm, a large pailful of cold water from an adjoining stream was dashed in with sufficient force to make the hot water run over, it being lighter; and this cooling process was continually resorted to. In this way the singlings were drawn off in about two hours, and those from four distillations made one charge of the still to produce the potteen.

The malt was prepared by inclosing the barley in a sack, and soaking it for some time in bog water, which is deemed the best; then withdrawing and draining it for some time, after which it is made to germinate in the ordinary way. When it has grown sufficiently, it is conveyed in a sack to the kiln. Besides the much-valued flavour of potteen, it derives a part of its character from being distilled entirely from malt. Frequently, however, about one-fourth of raw corn is added. From a bushel of this mixed grist the potteen maker obtains a gallon of spirit, of what he call three-to-one; or three glasses of spirit mixed with one glass of water afford proof spirit. This is, according to calculation, much below the produce that ought to be obtained.

The body of the still cost £1, its head 4*s.*, the worm cost £1 5*s.*, the mash tun and flake-stand, 12*s.*; £3 was, therefore, the value of the whole distillery. Sometimes, however, they are constructed on a more extensive scale. It is very doubtful whether the aroma depends on the turf smoke, for it is stated that the spirit has the same taste and odour when coal is burned under the kiln. It is possible that the turf smoke may be absorbed by the spirit, for it is well known that there is a period of the alcoholic fermentation at which odours are apt to be retained. The peculiar flavour more probably arises from the bog water in which the malt is steeped. When dried in the kiln after steeping, the heat is often sufficient to char the bog extract remaining in the malt; consequently, this would communicate an agreeable smoky aroma to the spirit.

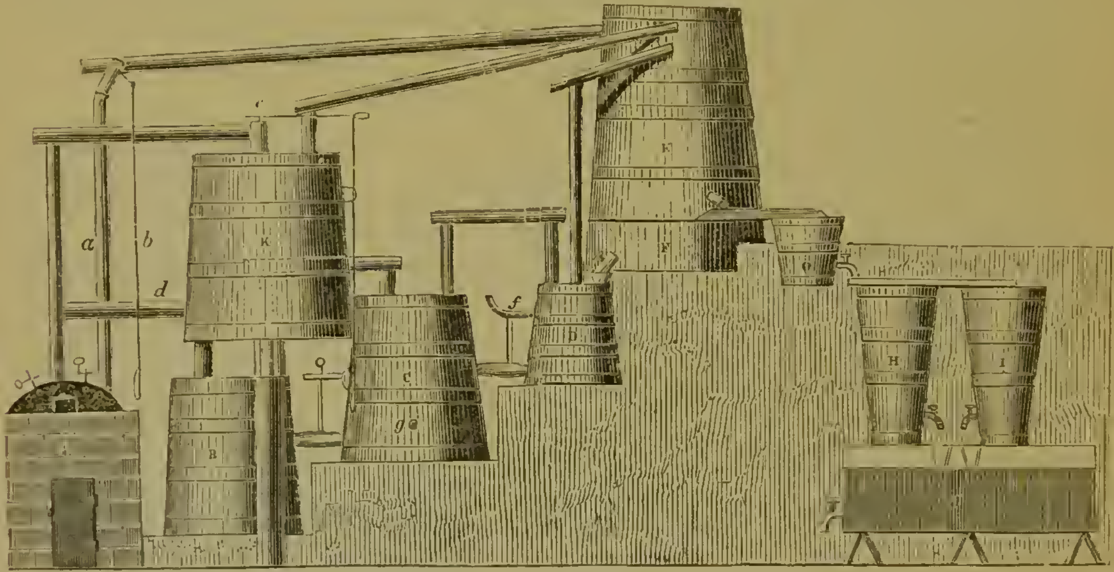
From a want of scientific knowledge and proper utensils, illicit distillers conduct their business in a different manner from that pursued by licensed traders. In preparing the malt, the sacks of barley are generally steeped in bog-holes or other places, where they remain forty-eight hours, or until completely saturated with the water. They are then drawn out and drained for ten or twelve hours. After this the grain is spread out upon the floor in a thick layer, and remains so till it begins to chip or germinate; it is turned occasionally, until all appears alike sprouted. It is afterwards spread by degrees, till such time as the buds show three points, and when these points have grown half way down the grains, by means of a regular heat, the particles are semi-transparent. At this stage it is spread thicker on the floor, and brought to a heat easily perceptible to the hand, then thrown into a round heap, and suffered to remain in that state for twenty-four hours, or longer; the latter is termed the rot or withering heap. It is then carried to the kiln and dried by turf; the kiln-head on which it is dried is covered with decayed straw, over which, if convenient, is placed haircloth or matting. The period of drying a kiln-head or crop, as it is termed, is commonly twenty-four hours, when directed by a person of experience. The grain, while on the kiln, is carefully turned, to expose every particle to the same heat, and to prepare it for coarse grinding. It is next taken to the still-house, which is usually a hovel or excavation near a running stream, or where there is a full supply of water. The quantity of malt to be brewed is commonly from 16 to 17 stones; after being bruised or mashed in the ordinary way, it is covered in the kiln with a lid or sacks, and suffered to repose for three or four hours. The worts are then drawn off, and cooled to a temperature regulated by the finger, no instrument being used for that purpose, and commonly to the same degree as that which is observed in regular distilleries; they are next put into a pipe or puncheon, with about a gallon of yeast; in an hour or two after the barm is added, fermentation begins, and in twenty-four hours afterwards the attenuation is considered complete. Sometimes two brewings, after undergoing the fermenting process for about eighteen hours, are considered fit for the still; and in the ordinary course of working a brewing is made every morning. The quantity of pure spirit drawn from these two brewings is usually 223 gallons, of one-to-two, or two-to-five; or, in other words, the spirit is of such a strength that it will bear 1 gallon of water to about 2 gallons of spirit, or 2 gallons of water to 5 of spirit, to bring it to proof. The usual strength at which illicit spirit is made, is from 4 to 6 over proof on Sikes' hydrometer; but sometimes it is as much as 8 per cent., and in many cases it has been sold at a strength of 30 over proof.

The flavour is entirely caused by the malt, and the mode of distilling.

In distilling the wash, the strong low-wines are separated from the weak, the latter being thrown back into the still with the succeeding charge of wash; a similar practice is observed in making spirit, the faints being put into the still with the next charge of low-wines. Thus the spirit is preserved pure and clear, nothing whatever being used in the distillation but a small quantity of soap thrown into the still with the pot-ale or refuse, to neutralize or keep down the yeast, as they term it, which would otherwise cause the run of the low-wines to become coloured like the wash, or to get foul. It is a mistaken notion to suppose that soap is used only by the larger distillers, since it is considered an indispensable article by every person who understands the mode of working a still on the old system.

Whisky is generally made in the United States from Indian corn; and in Cincinnati, where this grain abounds, there is so large a quantity manufactured as to supply the Western and Southern markets. In Pittsburg, and other parts of the United States, the whisky is purified by filtering it through charcoal coarsely ground.

Fig. 40.



Numbers of distilleries are now at work in Canada; they are mostly of wood, and worked by steam. The annexed drawing and description, Fig. 40, is of one near Toronto.

A is the brickwork, in which the iron boiler, with a cylindrical flue running through the centre, is partly inclosed. B and C are the first and second wooden stills, of the same size, being 4 feet 8 inches at bottom, and 4 feet 6 inches at top, with an altitude of 6 feet; D is the doubling or low-wines' still, 2 feet 10 inches at bottom, and 2 feet 4 inches at top, the height being 3 feet 9 inches; E is the worm-tub, 6 feet at bottom, 5 feet at top, and 9 feet high, supplied by a copious stream of water; F the low-wines' and faints' receiver; G is the recipient for the spirit previous to passing through the rectifiers or filtering vessels. H, I, and is 2 feet at bottom, 2 feet 4 inches at top, by 2 feet in height. The top diameter of H and I is 3 feet, and the bottom 2 feet, the altitude being 5 feet; they are filled with charcoal and other material, through which the liquor gradually descends in a limpid, gently-flowing current into J, the final receiver, or store cask. K is a tank or large vessel for holding warm water for distilling purposes, supplied from the top of the worm tub, the heat of which is supported by steam from the tube, *c*, connected with the boiler, and having a stopcock for regulation at *e*. The tank is a reservoir for supplying the mash tubs with water, of which, in the establishment, there are fourteen, each measuring 3 feet 4 inches by 3 feet 6 inches in diameter, ranged on a loft above the stills, in such a manner that, after the worts have undergone fermentation in these tubs, they are let down by a leader or trough into the second still, C, at *g*. When the first charge is worked off, the remainder is let into the first still, and the second still is charged from the mash-keeve. To facilitate the operation, there are pipes with proper stopcocks from still to still, such as that at *f*; and it will be perceived that the whole process of distillation is effected by means of steam admitted through the tube, *d*, projecting from the main upright pipe of the boiler into the first still, B, and so proceeding by other pipes through the other stills. The tubes which convey the steam into the stills descend to within 3 or 4 inches from the bottom.

All the vessels and pipes, as well as the stills, are made of pine; the pipes are 9 inches square, with a bore of 2½ inches in diameter. The steam-boiler is 7 feet deep, the height of which, at the fire place, is 8 feet, and it is supplied by water from the worm-tub by the pipe, *a*, regulated by a stopcock or ball of lead which is worked by the cord, *b*. It is not necessary to describe the other vessels of this arrangement, as they are similar to those employed in the distilleries of Scotland and Ireland. The greatest disadvantage attending it, is the liability of the timber vessels to become unserviceable when the operations are discontinued for any time; but in a country like Canada, where wood is plentiful, this inconvenience is readily repaired.

The wash is usually made from rye, wheat, or Indian corn, with a mixture of one-twentieth part of barley malt, or one pound to the bushel of mixed grain; many use a larger quantity. This is ground or crushed in a mill, and then mashed with water, at a heat ranging between 158° and 162° Fahr. (70° and 72°·2 C.); others work at a temperature so high as 180° and 190° Fahr. (82°·2 and 87°·7 C.), but this is unusual. When mashed, a cover is immediately put on the tubs, or keeves, in order to keep in the heat as much as possible. The mash is then permitted to remain, with an occasional agitation by the rakes for about two hours, until it acquires its proper sweetness; at this stage cold water is added to reduce the heat to between 70° or 74° Fahr. (21°·1 to 23°·3 C.) when yeast is added. The tubs or keeves are again covered and allowed to repose until completely fermented, when the distilling commences. The whole mash, grains and all, are put into the still. Brewing and distilling are generally carried on in the Canadas from October till May.

RECTIFICATION OF SPIRIT.—On recapitulating the steps through which the production of spirit has been traced, it will be seen that first the grain, or grain and malt, is crushed, in order to allow hot water to act more readily upon it; next, the mashing takes place and worts result; then the fermentation of the worts commences, and the saccharine matters are resolved into alcohol; finally, this alcohol is, by successive distillations, separated from its greater portion of water, and *plain British spirit* is obtained. Pure alcohol, the various forms of spirituous liquors known as hollands, whisky, gin, British brandy, and rum, and the cordials under the name of peppermint, cloves, aniseed, &c., are produced by the rectifier, from the plain spirit purchased from the distiller. British spirit in the form in which it leaves the distillery is but little known.

The manufacturer of whisky, or any of the other alcoholic liquors, rarely purifies the products, but disposes of them to the rectifying distiller, whose business it is to remove from them those contaminations which render them disagreeable or injurious. If, however, the fabricator conducts this operation as well as the original preparation from the grain, a distinct part of his premises has to be allotted to the work, to comply with legal enactments. The peculiar process followed will be here considered.

All spirituous liquors, when the extraneous bodies from which such liquors are obtained have been removed, are identical, with this exception, that a variable amount of water is present in them; they are all more or less concentrated solutions of alcohol; thus, the alcohol from wine, rum, malt, potatoes, carrots, beets, grasses, and various other sources, is the same in quality, provided all the other solid and liquid impurities be removed. A single distillation, however, will not effect purification, and when volatile oils are present, distillation only, how often soever repeated, will not separate them, for these volatile impurities pass over during distillation; hence, the spirit procured from wines by simple distillation will have their peculiar flavour; beer, when distilled, will for the same reason yield alcohol possessing the abominable taste of the yeast; malt spirit will have the disagreeable qualities of faints from the presence of an oil of an acrid bad taste; and potato spirit the physical characteristics of fusel oil, or oil of potatoes. Thus the disagreeableness or fragrancy of distilled products is, as in the case of malt and potato spirit, due to the presence of an essential oil, derived from the source of the alcoholic liquid. The chief object of the distiller in rectifying spirit is the removal of these oily bodies in order to procure a pure alcohol, from which, by the aid of other ingredients, he can fabricate liquors imitating those more costly products which are formed naturally, such as the better varieties of brandy, gin, whisky, and all the other kinds of liquors and cordials which are in daily request as favourite beverages with the community.

The cognacs, genevas, whiskies, &c., fabricated in this country are all made from the low-wines of the malt distiller, or from the potato or carrot spirit of foreign manufacturers.

All of these spirits, as they come from the first distillation of their respective mashes, are called low-wines or *singlings*, and being charged with oils, are unfit for any use while in that state. When they are redistilled, the first portion which comes over is much purer, and contains less water than the singlings; this is called the *foreshot*. So much of this spirit as can be produced without the smell of faints, is retained as whisky, and the remaining portion is diluted and submitted to further distillation.

The strength of whisky, as sent out from the manufactory, is generally about 25 per cent. over hydrometer proof, on Dicas' scale. On a further distillation of this whisky, a spirit is obtained which marks about 46 per cent. over proof, and is about the strength of spirit of wine in general; of this, about 80 gallons are obtained from 100 gallons of ordinary whisky, of 25 per cent. over proof. A further distillation affords a liquid of spec. grav. 0·820, which is the strongest spirit that can be obtained by mere distillation.

The presence of fusel oil in alcohol may be recognized by its taste, especially after dilution with a large quantity of water; and by its smell, particularly after rubbing the alcohol between the hands, or allowing part of it to burn away. **VÖGEL** finds that alcohol to which nitrate of silver solution has been added, when placed in the sunshine, remains perfectly bright and clear; whereas, if it contains fusel oil, it becomes very strongly reddened.

GÖBEL states that the origin of any particular alcohol can be ascertained by the following means, even when no particular odour can be detected by ordinary examination. A solution of 1 part of potassium hydrate in a small quantity of water is added to 160 parts of the brandy or spirit to be examined; after agitation the whole is reduced by slow evaporation to 15 parts. An equal volume of dilute sulphuric acid is then added, and the mixture well shaken. In the case of potato spirit the vapour evolved has a very disgusting odour, and produces when inhaled spasmodic contraction of the throat, headache, and giddiness. In the case of malt spirit the odour resembles that of sour dough, and produces a similar though less powerful physiological action. Rum, arrack, brandy, have when treated in this way each their characteristic and perfectly different odour.

Caustic potassa—under the name of grey salts—or soda, is added to unite with the oil; pearl-ash—white salts—is employed with the view of combining with the water, as well as to take up the fatty or oily impurities. The alkali combines with the oil, giving a soap which remains in the still, while the spirit passes off divested of its impurities. It has been ascertained that when carbonated alkali is used, the distillate contains a sufficient amount of the salt to affect reddened litmus and turmeric; hence this method cannot always be followed.

When calcium chloride, caustic lime, sodium sulphate, potassium acetate have been employed, either for the purpose of uniting with water or oily matter in the liquors, portions of these bodies are, according to the statements of **DUBUC**, invariably found in the distillate. The same authority states, that when spirit was distilled over calcined alum, the condensed liquid reddened litmus paper; but when sulphuric acid was added in small quantity to the spirit, the product which came over was found to be quite pure. Alumina, or aluminous clay, well washed and dried, was found capable of extracting small quantities of water from spirit, and no portion of it passed over into the receiver.

Charcoal is a means by which the disagreeable flavour of spiritnous liquors is removed. It has been made the subject of investigation by many chemists, among whom **LÖWITZ** ascertained that, by distilling common malt spirit over charcoal, or by merely agitating the two together, its peculiar bad smell was removed. He performed ten experiments with one pound of common spirit for each operation, and a variable quantity of charcoal, from half a drachm to five ounces, with the following results:—

Half a drachm of charcoal scarcely altered the smell, and the liquor did not clear for six months.

One drachm of the charcoal had no better effect in removing the bad flavour and odour; but cleared it in four months.

Two drachms cleared the spirit in one month.

Four drachms removed the smell in a very sensible degree, and the spirit became clear in one month.

One ounce of the charcoal completely freed the spirit from the bad smell, and clarified it in a fortnight.

An ounce and a half cleared it in eight days; three ounces in five days; four ounces in twenty-four hours; and five ounces in two hours.

The charcoal, mixed with water and distilled, yields an additional quantity of alcohol, but very impure, because at the heat required for the distillation part of the absorbed fusel oil is likewise parted with. The charcoal is said to be the more active the softer it is. For purifying brandy **STICKEL** prefers bone charcoal to wood charcoal.

KUNKEL was the first chemist who made the essential oils contained in spirit the object of research. His method of rectification was to dilute largely with water and distil. The fusel oil being less volatile, passes over towards the end of the distillation, and yields, when nearly all the alcohol has passed over, a turbid distillate, because the alcohol which distils over at that stage of the process contains too much water to hold the fusel oil in solution. The head and condensing worm must be cleaned after each distillation, or otherwise the alcohol which passes over at the beginning of the following distillation will be strongly contaminated by the fusel oil deposited in them.

Wood-ashes had for a long time been almost in general use in the rectification of spirituous liquors; their beneficial effects are due to the amount of alkali they contain, which enters into combination with the small quantity of acid formed in the spirit, as well as with the oily matters.

It would appear that filtration through hydrate of lime has the effect of removing some of the essential oils from spirit; the practice had been followed in France, particularly with those who carried on an illicit trade by adding an odoriferous oil to their brandies, and then entering them at the customs as perfumes.

The fragrant principle was removed, subsequently, by diluting with water, and filtering through lime, slaked in the air, which had the effect of separating the oil, provided it had not been present in great excess.

ZEIZE affirms that agitation with a small quantity of hypochlorite of lime (bleaching powder) has the effect of destroying the essential oil, and that the spirit, after distillation from the lime compound, resembles brandy in flavour. To 1 part of bleaching powder, previously triturated to a paste with water, he adds about 664 parts of the spirit to be defuselized, and distils after forty-eight hours. If the quantity of bleaching powder is too small, some fusel oil escapes decomposition; if too large, other products, notably chloroform, pass over.

Milk, according to the views of M. Cadet de Vaux, has the peculiar property of removing the bad flavour from the spirit obtained from the wines of various mellow and sweet fruits. This is effected by one or two distillations, and the spirits obtained are almost identical, notwithstanding that they were procured from quite different sources.

The rectifying distiller has now only to remove the essential oils, since the improved apparatus used affords an alcohol of any strength required for ordinary purposes. The proportion of salts employed for removing the oil from the crude proof spirit of the large wash distillers, is about

4 lbs. grey salts,
4 lbs. white salts,
700 gallons plain spirits.

When the distiller infers that an unusual quantity of foul oil is present, the proportion of salt is increased, and sulphuric acid is also added in order to improve the flavour of the spirit by giving rise to an ether. The salts are dissolved in about two gallons of liquid, and the sedimentary impurities removed by filtration. The solution is then mixed with the crude spirits, and distillation commenced. If the common still be employed, great attention must be paid to the fire; for if it be not slackened when the liquid first boils, there is much risk of the still *running foul*, that is, boiling over through the neck of the still, which occurrence would of course spoil the operation; when, on the contrary, the improved stills, which have been already described, are used, no danger is to be apprehended from this evil.

In rectifying faints, 80 under proof, the following method is used:—The spirit is mixed with the proper quantity of alkali, and the stills charged; the first portions that come over are collected separately, till the spirit runs at about proof, when it is turned off into another receiver; the result of this rectification —the common still being employed—will be about 10 per cent. over proof. On rectifying a second time, the distillate at first marks 43 per cent., and is collected till it indicates 30; it is then conducted into another back until reduced to 10 per cent. over proof, and the residual portion, after this strength, is received in the faints-back. A third rectification will afford a spirit of 53 per cent. over proof, or spirit of wine. In the second rectification, only one half the quantity of salts employed in the first operation is made use of; and in the third, it is customary to add about 4 lbs. of animal charcoal, and 10 lbs. of coarse grained common salt, to every 100 gallons, to cleanse the still. When St. Marc's stills are used 1200 gallons of proof ordinary wash spirit can be rectified in ten hours, product being 60 per cent. over proof, and stronger; a longer time is required to distil the whole contents, but the trouble of repeated distillations is dispensed with, besides, a considerable saving in labour and fuel is effected.

Geneva.—This liquor has been long the subject of study among distillers. Many trials have been made to produce in this country a spirit equal in quality to that imported, but with very indifferent success.

The subjoined particulars were communicated to the late Dr. Thomson, by a gentleman who resided in Holland for several years, solely for the purpose of learning the process followed in the manufacture of the Dutch hollands or geneva.

112 lbs. of barley malt, and 228 lbs. of ryemeal, are mashed with 460 gallons of water, at 162° Fahr. (72°·2 C.); after infusion has taken place, cold water is added to bring the strength to 45 lbs. per barrel, or spec. grav. 1·047, at which strength, after it has cooled to 80° Fahr. (26°·6 C.), it is run into the fermenting tun. To the contents of the fermenting back, which is about 500 gallons, half a gallon of good yeast is added; fermentation speedily sets in, the temperature rises to about 90° Fahr. (32°·2 C.), and the attenuation is complete in forty-eight hours. After attenuation of the wash, from 12 to 15 lbs. per barrel of saccharine matter remain undecomposed in the fermented liquor. The wash and grains are then introduced into the still, and the whole of the low-wines distilled over; these are subjected to a second distillation, and the distillate after rectification is the famous "geneva." A few juniper-berries and sometimes hops are added in the rectification, to impart to the spirit its peculiar flavour.

Some peculiarities may be noticed in the process followed in Holland, namely, the imperfect attenuation of the wort and the small amount of yeast employed in bringing it about. The British distillers obtain double the quantity of spirits from their worts that is produced by the Dutch distillers, if the example just given be true. It is very probable that the large amount of yeast used by British and Irish distillers, and the efforts made to effect as low an attenuation as possible, are the causes of the flavour of British gin being so inferior to Hollands. The only liquor in this country which can bear comparison with that of Holland is the illicit product. A manufactory for the production of Hollands was instituted some few years since at Maidstone, in Kent. It never became popular, and after languishing a few years ended in bankruptcy. It has never since been revived.

British Gin is, for the most part, manufactured by the rectifiers of the low-wines of the Scotch and English spirit or whisky fabricators. These processes are generally carried on in a somewhat uncouth fashion.

The still is charged with 300 gallons of liquor, and 650 gallons of spirit from a previous rectification, to which are added:—

95 lbs. German juniper berries,
95 lbs. coriander seeds,
47 lbs. crushed almond cake,
2 lbs. angelica root, and
6 lbs. liquorice powder.

The whole is well rummaged, distillation commenced, and after the worm is cleansed by the first portions drawn over into the faints-back, about 160 gallons are run into forcing-back No. 4, then turned off into back No. 3, till it runs 1 to 9, or 11 per cent. over proof, when it is turned into faints-back No. 8.

About 400 gallons are found in back No. 3. Liquor is run into back No. 4 to reduce it to 50 per cent. under proof; it is fined by throwing into it 2 lbs. of alum dissolved in boiling water, and leaving it to rest for about eight hours, after which this *low gin* is pumped into back No. 3, containing the remainder of the charge, to bring it to 22 per cent. under proof; then the whole is pumped into store casks for use.

Another standard recipe for *Cordial Gin*:—Take 700 gallons of the product of the second rectification (if the improved stills are used the product of the first distillation answers), and mix with it the following ingredients:—

70 lbs. German juniper berries,
56 lbs. coriander seeds,
5 lbs. almond cake, crushed or broken,
1½ lb. orris root, broken,
2½ lbs. angelica root, cut,
½ lb. cardamom, or, instead of this,
6 lbs. liquorice powder are sometimes added.

Force the first running of the working, or about 200 gallons, by reducing it to 50 under proof, adding three quarters of a pound of alum, boiled in 2 quarts of water. In adopting this recipe, make a double working of it with twice the quantity of the ingredients. Work in the flavouring in the first charge of rectified spirits, having in the back 2 or 3 inches of the usual charge, to make up with liquor, and prevent the bottom of the still from injury by the charring of the large amount of ingredients depositing upon it. Turn the distillate into another back, and reduce to 50 per cent. *under proof;* force with 1½ lbs. of alum and pump into fining cask; then charge with rectified spirits and work into back containing goods from preceding charge. Run down gin from store cask and make up to strength required—17 to 22 under proof.

Another recipe for the manufacture of Cordial Gin, the charge being 950 gallons, is the following:—

100 lbs. juniper berries,
70 lbs. coriander seeds,
2 lbs. orris root,
1 lb. angelica root,
2 lbs. calamus root,
½ lb. cardamom seeds.

The operations being the same as those just recounted.

For a *Fine Gin*, the distillation of which is carried on for the most part in the same manner as the above, take

960 gallons of spirit, hydrometer proof,
96 lbs. German juniper berries,
6 lbs. coriander seeds.
4 lbs. grains of paradise,
4 lbs. angelica root,
2 lbs. orris root,
2 lbs. calamus root,
2 lbs. orange peel,
80 or 90 lbs. of liquorice powder are occasionally added, to impart colour and sweetness.

Plain or *London Gin* is made in the following proportions:—

700 gallons of the second rectification,
70 lbs. German juniper berries,
70 lbs. coriander seeds,
3½ lbs. almond cake,
1½ lb. angelica cake,
6 lbs. liquorice powder.

For the manufacture of *West Country Gin* the annexed is the process:—Introduce into the still 700 gallons of the second rectification, and flavour with

14 lbs. German berries,
1½ lb. calamus root, cut, and
8 lbs. sulphuric acid.

This gin is much used in Cornwall, and particularly in the western counties of England; it is also used in making British hollands, and in that case is mixed with about 5 per cent. of fine gin, reduced to 22 under proof with liquor.

It may be observed that all these processes merely consist in flavouring rectified spirit with various aromatic substances.

For *Geneva*, charge of still being 950 gallons of second rectification, the proportions are:—

84 lbs. juniper berries,
112 lbs. coriander seeds,
6 lbs. cassia buds,
4 lbs. angelica root,
6 lbs. calamus root.
6 lbs. almond cake,
½ lb. cardamom.

Plain Geneva.—For 950 gallons of spirit of second rectification, take

84 lbs. juniper berries,
84 lbs. coriander seeds,
2 lbs. almond cake,
2 lbs. orris root,
2 lbs. calamus root.

Another prescription for making Geneva—and one which is much esteemed—is the following:—Add to 950 gallons—

14 lbs. grey salts, and
4 lbs. white salts.

The rectification to be conducted with the usual care. At the second operation, add—

168 lbs. juniper berries,
74 lbs. coriander seeds,
12 lbs. almond cake,
8 lbs. grains of paradise,
8 lbs. angelica root,
1 lb. cardamom,
2 lbs. calamus root.

In making gin with a high degree of flavour, the distillate, or flavoured liquor drawn over, is very apt to turn blue on being diluted, to prevent which is very often a difficult task. In preparing such liquors it is therefore the most advantageous method to have two stills, and to divide the charge between them. To one-half the amount, the whole of the spices and flavouring matters are added, and the liquid drawn off into two backs till it runs at 11 over proof. One of the backs is reduced as low as 1 in 2, or 50 per cent. under proof, which ought always to be observed, as in making gin it should on no account exceed this strength, but may be 2 or 3 per cent. under, without being disadvantageous. About 2 to 4 lbs. of alum are dissolved in hot water, and this solution is thrown into the back with the flavoured spirit or goods, the whole briskly stirred, then pumped into a store vat, and left to fine over night; in the morning it will be clear, and is run into the back containing the second portion of the rectified spirit, without flavouring, and diluted to 22 or 17 per cent. under proof.

In making gin of a less flavour, or where two stills cannot be employed, it is necessary to have a forcing-back to fine. To this forcing-back a small cask is attached, to enable the operator to pump off the low gin to the portion of strong in the back. The process followed in order to prevent the *blueing* is to flavour and charge the still, and to run off into the forcing-back, till it is ascertained at the worm end that the spirit will not change colour on being diluted with water; when this happens, it is turned off into the working-back to the usual strength. The gin is then reduced in the forcing-back to 50 per cent. under proof, and the aluminous solution poured in; on the following day, when the liquor has clarified, it is pumped to the making vat to the remainder of the charge, and brought down to the strength required. If too weak, it may be raised by adding the best spirit of wine in the required quantity.

In France, an exceedingly fine gin is produced by fermenting a portion of juniper berries bruised with 4 parts of barley meal, or ground malt, proceeding throughout the succeeding part of the operation in the same way as when making grain spirit.

Another method practised is the following:—Boil during half an hour 2 gallons of bruised juniper berries in 4 gallons of water; put this into a barrel capable of containing 6 gallons; add to it at first 4 pounds of rye bread that has been dried and reduced to powder, then some aromatic, according to the fancy of the manufacturer, and 2 lbs. of brown sugar. At the end of a month the liquor is converted into an agreeable wine, which, when distilled, affords a spirit much esteemed, and commanding a high price.

Having given the several mixtures used by the rectifiers in preparing their gins, a notice is given below of each substance mentioned, with a description of the oil derived from it, and its properties.

Cardamom.—The oil obtained from this seed is colourless, of an agreeable odour, and strong aromatic burning taste; the seeds are used as a condiment and also in medicine; they are stimulant, tonic, stomachic, and carminative, and yield nearly 5 per cent. of essential oil.

Grains of Paradise.—The volatile oil has a light yellow colour, a camphor-like smell, and a hot, penetrating taste; the seeds are esteemed in Africa as the most wholesome of spices, and are generally used by the natives to season their food; they are also used for medicinal and other purposes, as stomachic and cordial stimulants. Gins possessing a very peppery taste acquire it from grains of paradise.

Almond Cake.—This is the residual cake of bitter almonds, after expressing the fixed oil. When distilled it affords a volatile oil, which has a golden yellow colour, an agreeable odour, and an acrid bitter taste. Substances or liquids flavoured with bitter almonds may act injuriously, owing to the presence of hydrocyanic acid.

Angelica Root.—This root grows most abundantly in northern Europe; its taste is at first sweet, then hot, aromatic, and bitter.

Calamus Root.—The taste of this root is due to the oil, which is sharp and sweetish; the yield of oil is about $\frac{1}{60}$th per cent. It is obtained by distilling with water, and is used for flavouring aromatic vinegar.

Orange Peel.—The peel of the orange yields the oil proper, which has the strong odour of the rind.

Lemon Peel.—The oil is obtained both by expression and distillation. It is chiefly imported from Portugal, Italy, and the south of France. Pure lemon oil is very fluid, of an agreeable odour, and colourless. Its taste is pleasant but pungent, and it is, therefore, often used in culinary purposes as a substitute for the peel; the flavour, however, which it imparts, frequently savours of turpentine, and is never so agreeable as that communicated by the fresh rind of the fruit. Oil of lemon and oil of turpentine are isomeric.

Juniper Berries.—What the distiller prizes in this berry is its volatile essence, which is an oily liquid, isomeric with the preceding and with oil of turpentine; their smell is also very similar, hence the origin of the supposition that London gin is sometimes mixed with the latter. Oil of juniper is the most powerful of all diuretics. It promotes perspiration and relieves flatulency; consequently, gin is recommended in many diseases of the urinary organs.

Coriander.—The seeds have a strong smell, and medicinally are considered as stomachic and carminative; they are used in sweatmeats, in certain stomachic liqueurs, and in some countries in cooking. They contain about half a per cent. of volatile oil, to which they owe their fragrancy, and on this account the seeds are used in rectifying operations.

Orris Root is the root of *Iris Florentina*, a white flowering species of iris found in the South of Europe. It has an agreeable odour resembling violets, and is used in perfumed powders.

Liquorice.—A plant of the genus Glycyrrhiza. The root abounds with a sweet juice, much used in demulcent compositions.

Cassia.—This plant furnishes buds which consist of the calyx surrounding and nearly encircling the young ovary. They bear some resemblance to a clove, but are smaller, and when fresh have a rich cinnamon flavour. They are used for the same purposes as cinnamon and cloves. The bark and oil are powerful stimulants. Oils of cassia and cinnamon on exposure to the air rapidly absorb oxygen, yielding a considerable quantity of cinnamic acid.

Most of the roots or seeds have, so far as their oils or essences are concerned, very analogous properties and since their virtue in distillation with the spirit is to communicate their oily or fragrant principle, it would be better to add a few drops of each of the oils at once to the pure spirit, to procure the desired liquor, and by this means obviate the necessity of distilling, and the risk of injury to the stills by the mixtures mentioned, which appear to have no atomic rule for their basis.

Brandy.—This liquor is generally obtained from the high-coloured, white, or pale-red wines of the south of Europe, but is often manufactured from inferior articles, such as the refuse wine and the marcs of the wine press.

Distillation of the wines is the only thing necessary to procure this spirit; hence, the richer the wine in alcohol, the greater will be the yield of brandy. A simple test with an alcoholometer will determine the value of the wine as to the yield of brandy; but many other circumstances, independent of the manufacture, enhance the quality of the product.

Thus, the white wines do not always afford more alcohol than the red, though the spirit from the former is of a much finer quality, the reason being that they contain more of the essential oil of the grapes. It is also a singular fact, that those wines which carry with them a certain taste of the soil, communicate it to the brandy derived from them by distillation; thus, the wines of Selluel, in Dauphiné, give a brandy which has the flavour and the taste of Florentine iris; those of St. Pierre, in Vivarais, give a spirit which smells of the violet, and so of many other varieties.

Wines from the countries nearest the Mediterranean furnish the largest proportion of brandy. The wines from the south of France yield one-fourth of brandy, some even one-third, while in the north of France the amount is only about one-eighth.

The better qualities of brandy are invariably distilled from white wines; first, because a greater yield of brandy, and of a better quality is obtained; and secondly, because those wines fine more quickly, and can be distilled into brandy sooner than the red wines.

The principal stills employed on the Continent are those of Champonnois, Derosne, St. Marc, and Laugier, in France; of Pistorius, and various modifications of his apparatus, in Germany. With these stills, the spirit comes over of any requisite strength, up to the strongest spirit of wine; but if the ordinary still is employed, the receiver is changed when the vapour arising from the boiler ignites but feebly, and the *eau-de-vie-seconde*, or *repasse*, is collected by itself, till the whole of the alcohol in the wine has been exhausted. The liquid remaining in the still is called *vinasse*.

The *campaign*, or distilling season, in France, is from the beginning of October to the end of May.

The following is an average of the yield of brandy which some of the wines afford by distillation:—

1000 litres of wine of St. Gilles, in the environs of Montpellier, afford of 3/6 brandy,....	150	litres.
" " of good wine of calcareous soils,......	140	"
" " of wines of fertile soils near Montpellier,	100	"
" " of wines of soils producing much grapes,	100	"

Payen gives the following table of the proportions by volume of pure alcohol contained in 100 parts of various kinds of wine:—

Port and Madeira,......	20	Bordeaux claret,.. . ..	13
Xeres, Lacryma Christi,	17	Barsac, white,... . ..	13·7
Old Madeira,..........	16	Poundensac, red, . ..	13·7
Jurançon, white,... ..	15·2	Blaye,..... . . .	10·2
" red,.........	13·7	Libourne,...	9·8
Malaga,..............	15·0	Saint Emilion,.. ...	9·1
Frontignan,..	11·8	La Réole,..	8·5
Vauvert,..............	13·3	Cubzac,...	8·7
Hermitage, white,......	15·5	Giscours,....	9·4
Sauterne,......	15	Laroze-kirwan,.. .. .	6·3
Beaune,..............	13·5	Cantenac,.............	9·2
Volnay,..............	14·7	Volnay,...............	11
Dijon,....	13·0	Mâcon,....	10
Chambertin,.	12·4	Champagne....	11·6
Château Lafitte,.......	8·7	Saumur,.......... ...	9·9
Château Margaux,.. ...	8·7	Tokay,..	9·1
Château Latour,.......	9·3	Rhine,..	11·9
Saint Estèphe,........	9·7	Chatillon,....	7·5

Brandy, as sold in France, is generally of two degrees of gravity; these strengths are thus designated—*à preuve de Holland*, and *à preuve d'huile*; the former varies from 18° to 20° of Beaumé. The stronger liquids are valued according to the quantity of *eau de vie*, or brandy *à preuve de Holland*, that a given quantity will furnish on the addition of the proper proportion of water. These strengths are usually twelve, namely, *five-six*, *four-five*, *three-four*, *two-three*, *three-five*, *four-seven*, *five-nine*, *six-eleven*, *three-six*, *three-seven*, *three-eight*, and *three-nine*, but the last is rarely made. The meaning of these strengths is understood in the following sense:—If a spirit be five-six, five parts of the spirit will give a liquor *à preuve de Holland*, when added to six measures of water; if three-six, three measures when added to six of water will yield a spirit of the same standard, and so of the remainder.

The spirit five-six strength is of a specific gravity 0·9237, or 22° Beaumé; but all the other strengths are subject to variation, on account of the uncertainty of the strength *à preuve de Holland*, as before shown. Wines, when distilled carefully from a clean apparatus, yield a distillate which is nearly colourless, and when it is wished to retain it in this state, vessels of glass or stoneware are employed. After being put in casks and left in them, the clear liquor acquires a little colour by extracting the soluble matters of the wood; the flavour, however, is not materially affected if the casks are old; when new casks are used, the tannic acid of the wood, which is generally oak, communicates a deep colour and astringency of taste which is quite foreign to the brandy.

The brandy from different localities, and even from a different variety of grape grown on the same place, possesses, as already remarked, an aroma characteristic of the wine whence it is obtained, and which is readily perceptible to those well versed in the trade. An experienced taster will readily distinguish the brandies of Languedoc, Bordeaux, Armagnac, Cognac, Aunis, Rochelle, Orleans, Barcelona, Naples, &c.; further, he can say from what species of fruit it is derived; and he will also discern minute shades of difference in the qualities of various brandies from the same source. Real cognac is obtained from the distillation of the choicest wines, every regard being paid to the proper degree of cleanliness which should be observed in the various utensils through which it has to pass. In the improved forms of still a very superior article is obtained from inferior wines, but the large proportion of essential oils in such wines spoils the flavour of the brandy.

An inferior variety of brandy, or *eau de vie de Mares*, is obtained by distilling the dark-red wines of Portugal, Spain, and other wine-growing countries, the lees deposited by wines when kept, the mares or refuse of the grapes from the vine-press, the scrapings of wine casks, &c.

Distillation is with these inferior materials carried on in the ordinary way, but as the flavour is less regarded, the spirit is drawn off rapidly, and at a high temperature. The mares from the vine-press are prepared for the purpose of distillation by breaking the cakes up into pieces, and throwing them into water. A temperature of 70° to 80° Fahr. (21°·1 to 26°·6 C.) is kept up, and in the course of a short time fermentation sets in; when this has ceased, the solution is racked off and distilled. The first distillate has a whitish colour, and is called *blanquette*, but this on redistillation yields a spirit of 22° or 24° Beaumé. One pound of brandy is produced from 85 to 90 lbs. of cake.

The fermentation of the cakes is sometimes effected in large pits, where they are covered with earth. The progress of fermentation is known by thrusting the hand into the heap. When the temperature decreases, the fermentation is said to be finished; the contents are then taken out, water in proper quantity added, and distilled. By this process 100 lbs. of mares yield 1 lb. of brandy.

When such liquors are distilled, the sedimentary matter subsides, and is apt to carbonize on the bottom of the copper still; when it does so it communicates a smoky flavour to the distilled liquor, in addition to a hot fiery taste proceeding from the fusel oil of the skin of the fruit. AUBERGIER has proved that a few drops of fusel oil are sufficient to taint a pipe of 133 gallons of pure spirit.

In some distilleries the carbonization is hindered by keeping the contents of the still in motion by agitators. Other manufacturers insert into the body of the still a basket to retain the sediment accompanying the fermented liquor, thus preventing its contact with the lower part of the still. M. REBOUL'S process is to inject steam into the still by means of a coil of piping. The stills in this case are large wooden boxes, to which worms are adapted in the usual way, and the whole of the alcohol is driven over by steam heat. M. CURANDAU'S apparatus consists of a still, the neck of which is as wide as the body, and 3 feet in height; brackets are placed at the distance of 9 inches in the neck of the still, which support several partitions; these are provided with short pipes, pierced with holes, to allow the vapours to circulate freely from the body of the still. The neck being affixed, and the first partition introduced, wine lees are poured in, which are filtered by the perforated partition; then the second, and a further quantity of lees, and so on till all the partitions are inserted, and the lees are about 6 inches thick on each. If the liquid drained from the mares or lees be not sufficient to fill the still, water is added to make up the proper quantity. Heat is then applied, and the steam, as it passes off, expels the last portions of spirit from the solid particles retained by the partitions in the neck of the still.

Most of the inferior kinds of brandy contain an acid which partly unites with the oil from the grapes, rendering the taste of the spirit unpleasant; agitation with a little quicklime not only removes the acid, but also the oil in a great degree. The matter left in the still when dried and burned yields an alkaline carbonate, which is disposed of for dyeing operations.

Spirits of this class are used by the lower order of people in France, and on account of their hot fiery taste are often preferred in England and other northern countries to a more genial produce. Cognac and Armagnac brandies contain about half their weight of water, and owe their fragrancy to the aroma, indigenous to the wine, being less disguised by the fusel oil.

Ethyl pelargonate ($C_2H_5,C_9H_{17}O_2$), the ether of pelargonic acid ($C_9H_{18}O_2$), originally described as œnanthic ether, is a liquid possessing a most powerful and intoxicating odour. The aroma of wine is in great measure due to the formation of this ether during fermentation. When wines or mares containing this ether are distilled, an oily liquid passes over towards the close of the operation, which consists in great measure of crude ethyl pelargonate, and imparts the aromatic odour which cognac and other liquors possess. PELOUZE first proved this odorous principle of wine to be a compound ether. MULDER detected the same in the oil of grain-spirit and in other fermented liquors, and it is from this ether that quinces derive their distinctive perfume.

The effect of heat on several of the substances contained in wines merits the attention of the distiller. A temperature slightly too high sometimes destroys a whole distillate, as empyreumatic and other products are generated.

When, to save expense in carriage, the spirit is rectified to a much higher degree than the above, the dealer, on receiving it at Paris, reduces it to the market proof strength by the addition of water or of a little fragrant weak brandy; but the brandy produced in this manner is not equal to that derived from the distillation of Cognac wine at an incipient heat. This may be readily proved by submitting to distillation, with every precaution, brandy of a superior quality; if the resulting spirit be then brought down to the ordinary strength with water, it will be plainly perceived that the liquid has been considerably deteriorated by the operation. Genuine French brandy evinces a red reaction with litmus paper, owing to a minute portion of acid; and when kept for a considerable period in casks, it acquires an astringency which impairs its quality.

The brandy sold in England is, for the most part, artificial—the fabrication of the rectifying distiller. The following is one of the recipes for the purpose:

—Dilute the pure alcohol to the proof pitch, and add to every 100 pounds weight of it from half a pound to a pound of argol—crude winestone—dissolved in water, some bruised French plums, and a quart of good cognac. Distil this mixture over a gentle fire, in an alembic provided with an agitator. The addition of brandy and argol introduces œnanthic ether, and if a little acetic ether be added to the distillate, the liquor acquires the peculiar taste of genuine cognac brandy; colour with burned sugar, if necessary, and add a little tannic acid to impart astringency.

The process followed by the rectifier is somewhat different, as will be seen from the subsequent few examples, which are transcribed from the private work-book of a very extensive rectifying distiller. The source is generally the common low-wines of the grain distillers, which are rectified in the usual way by distillation with caustic salts, as has been already described.

To every 500 gallons of this spirit, about 25 gallons of the best French wine-vinegar are added, and the whole well rummaged in the mixing-back; the mixture is then pumped into the still, and a further quantity of a weaker spirit run into the back, in order to clear it of the last traces of vinegar; this liquor is also pumped into the still to make up. From 56 to 60 lbs. of coarse-grained common salt are now mixed with the liquid in the still, together with from 8 to 10 lbs. of concentrated sulphuric acid, keeping the whole in brisk motion during the addition of the latter, to protect the still from the action of the acid. The fire is then lighted, and the still brought down and worked till the spirit shows a strength of 14 over proof, or one-to-seven. It is customary to turn off into the faints-back at a lower degree of strength, and collect the remaining quantity of faints; in such a case, however, the quality of the spirit is not so good. From every 500 gallons of the charging, 500 to 510 gallons of spirit, marking 42 per cent. over proof, are obtained. This is mixed with from 400 to 450 gallons of liquor, and pumped into the British brandy store-vat: 20 to 25 gallons of *fruit tincture*, 15 gallons of brandy flavour, and 8 to 10 gallons of good colouring are then introduced into the piece, the whole well rummaged, and left to fine. It is considered an improvement to fine with skimmed milk. Some distillers prefer to introduce distilled vinegar in the proportion of 15 to 20 gallons to 1000 gallons of the compounded liquor in the store cask, instead of adding it previous to distillation, as before mentioned.

Another recipe, which is followed for the most part in the distillery from which these details were obtained, using a multiple of the number, is next given.

Three hundred gallons of proof spirit are distilled with the proper addition of caustic salts, taking all the precautions mentioned in the preceding, under Rectification of Spirits, and the distillate is received into the spirit-back till it runs at ten over proof. The remaining spirit is turned into the faints-back, and is then made up with 450 gallons of spirit, 22 under proof, 20 gallons of *prune tincture*, 20 gallons of distilled vinegar, and 8 gallons of good colouring matter.

When flavoured faints are cleansed, charcoal is employed in the rectification with sulphuric acid.

Raspberry Brandy.—In manufacturing raspberry brandy, the subjoined is the process:—For 1000 gallons of the brandy, take

460 gallons of raspberry tincture,
115 gallons of cherry tincture,
240 gallons of sweets,
96 gallons of British brandy, 22 U.P.
89 gallons of liquor;

rummage the whole well, and force or fine with isinglass.

Cherry Brandy.—To make 1000 gallons, take

575 gallons of cherry tincture,
253 gallons of sweets,
92 gallons of British brandy, 22 U.P., and
80 gallons of liquor;

the whole to be well agitated, and fined with isinglass.

Raspberry tincture is made as follows:—Take a brandy puncheon with head out, and screen over the cock; put in 50 gallons of clean rectified spirit of 22 under proof, then fill the cask with raspberries. In three weeks or a month draw off the whole of the tincture into a clean cask, and add to the fruit a second time 25 gallons of spirit of 22 under proof; let this remain upon the raspberries a month longer, and then draw this off, and add it to the first tincture; after which the whole of the tincture remaining in the fruit is to be pressed out and added to that already obtained. The cake is then broken up and steeped in a rum puncheon with the head out, 40 gallons of spirits are added, the whole contents briskly agitated from time to time for three or four days, and pressed well after drawing off the solution. This liquor is employed in making up the raspberry brandy instead of water.

Cherry tincture is made in the same manner as the raspberry, by substituting the one fruit for the other.

For prune tincture cover 56 lbs. of prunes, thoroughly broken up, with 20 gallons of clean spirit of wine, and after being allowed to stand for eight or ten days, rack off; the refuse fruit is washed twice with liquor, and the residue is then thrown away.

Many rectifiers prepare a brandy flavour as follows:—To 100 gallons spirit—clean faints, 54 over proof—add

100 gallons of good strong vinegar,
4 gallons of spirit of nitre,

in a back, and mix the whole thoroughly; cover closely, and the next day run it into the still with

8 lbs. of nitric acid,
10 lbs. of almond cake,
5 lbs. of orris root, and
2 lbs. of lemon peel;

work the still slowly, and turn off at proof strength.

In making up brandy, 10 per cent. of the above flavouring is employed, but more or less may be used to suit the taste of the consumer.

In Switzerland brandy is distilled from the refuse of the grapes after the juice is pressed out. Large casks are filled with the skins, which are squeezed as compactly as possible, and covered closely to prevent the access of air; fermentation usually commences in about three days, and when it has finished, which requires a considerable time, the mass is deemed ready for the still. When the distillation is to take place, the fermented mass is mixed with a due proportion of water, to reduce it to a proper consistence for the action of the fire, which is moderately applied to prevent empyreuma. It is said that a vessel containing 32 cubic feet of this material will yield 10 gallons of pure brandy.

RUM.—Molasses is the name given to the syrup which remains after the crystallization of sugar; it is, in fact, the mother liquor of sugar. This syrup, diluted with a sufficient quantity of water, undergoes the vinous fermentation, and by distillation yields a spirit called in the colonies *rum*, or *taffia*; the name given to it in the Isle of France and Madagascar is *guildive*. This spirit is of excellent quality when prepared with proper precaution, and particularly relished when it is very old. The best rum is made solely from molasses. An inferior quality is made from the debris of the sugar-cane, which has always a sharp disagreeable acid flavour, and frequently acquires an empyreuma, on which account it is given to the negroes who work in the sugar-houses, and is consequently called *negro rum*.

In the fermented liquor from which the rum is distilled, acetic acid sometimes exists in large quantities, accompanying the ardent spirit without forming ether; but in distillation a certain quantity of acetic ether is formed, which, from its extreme volatility, rises in the first process of distillation, giving to the vapour a most disagreeable taste and smell; hence the colonial saying, that the rum becomes *too hot* if rectified like European spirits. The cause of this is easily explained: the rectified spirit only forms a part of the charge of the still, and contains, nevertheless, *all* the acetic ether.

Skilful distillers, who pride themselves on making these strong spirits most agreeable to the taste, take great care to remove all kinds of vegetable matter or refuse incapable of producing vinous fermentation, as such substances have a tendency to putrefy, and the putrescent matter retards the action, and gives a savour which is communicated to the distilled spirit.

The Chinese, who prepare the famous arrack of Batavia, which is the best of all rums, take much care in rectifying it, mixing with it during distillation a composition called *ragie*, in which is cinnamon and anise-seed, in such proportion as not to be perceived either by smell or taste, being only sufficient to do away with the otherwise nauseous odour of the liquor. The Madagascars throw in leaves of trefoil. The Asiatics mix with it the bark of a kind of thorny acacia, called *pattay*. Some persons put into the still with the grape the leaves of a tree named *attier* in the East Indies, and *pommier cannelle* at St. Domingo—*anona squammosa*—which have a light agreeable odour. Others have tried with success the mixture of peach leaves. All these substances impart to strong liquors a pleasant bouquet and taste, and are used to disguise the smell of the spirit, and to give it unctuousness.

The chief seats of the distillation of this spirit are the East and West Indies, America, and France. It is strange that the Chinese, who produce so much sugar annually, and who, consequently, might manufacture large quantities of rum from molasses, have not hitherto attempted to distil this article.

In the West Indies the liquor of the sugar-cane runs warm from the coppers through a trough to a receiver prepared for that purpose. It is then skimmed, and the skimmings, with some of the liquor itself, are pumped into a cistern containing from 300 to 800 gallons, when the fluid is mixed with water and molasses in the proportion of 25 gallons to 100. When this mixture is sufficiently blended together in the vats, which in some plantations amount to thirty, it is covered over with boards or mats of plantain leaves, and allowed to ferment for three or four days, or longer, should there be a want of yeast or other ferment to make it work, which often occurs at the commencement of the season. When reduced to a due degree of acidity, which is ascertained by the subsidence of the fermentation, it is run into a still proportioned to the vat, and wrought off as low-wines, in which state it is put into the still again. The first run, or discharge, after it is thus returned to the still, is taken off for *high*-wines, as they are termed, or strong rum, in the proportion of 25 to 300 gallons, the strength of which, when tried by a glass-bead instrument, is from 18° to 22°. The second run of the still, which is drawn off in cans, and carried by negroes to another vessel, is of a strength from 23° to 26°. From these two runnings of the still the rum exported from the colony of Demerara is made up. Any deficiency in the strength of the second distillation is compensated by an addition from the first, which is always stronger than that exported, and of too ardent a nature to be used by itself, 25° being colony proof.

In the Windward Islands, one-third of the skimmings is mixed with one-third of the lees and one-third of water. When these begin to ferment, which they usually do in twenty-four hours, the first mixture of molasses is made in the proportion of 6 gallons for every 100 gallons of the fermenting liquor, and a day or two afterwards an additional quantity of molasses is added. The fermentation is tempered by the addition of cold or warm water. *Dunder* is the lees or feculencies of former distillations, and serves all the purposes of yeast in the fermentation. It is derived from a Spanish word, *redundar*, the same as *redundans* in Latin, and is well known among the planters in the West Indies. The attenuating properties of this ferment are such, that the materials with which it is mixed are said to yield a much greater proportion of spirit than could be obtained if they were fermented without it; it serves the same purpose as jalap mixed with molasses, which has been sometimes employed in Great Britain for cutting down the frothy head at the close of fermentation; and it is usually preserved from one year to another for this purpose, in such large quantities as to fill most of the backs or fermenting tuns. Dunder soon becomes covered with so thick a film as to exclude the air, and the sediment leaves the intermediate fluid pure, of a bright amber colour, which, when carefully drawn off, is employed as already described, in proportions suited to the nature of the fermentation.

Dunder fulfils two important offices in the distillation of rum. In the first place, the large quantity of acetic acid contained in it, and formed at the expense of alcohol during fermentation of the wash, serves to decompose the calcium saccharate contained in most West India molasses in considerable quantities. It is for this reason that dunder increases the yield of the rum. Saccharate of lime does not ferment, and when present in considerable quantity actually opposes the fermentation of the wash; so that without dunder fermentation would proceed so sluggishly, that most of the alcohol would be converted into vinegar. This occurrence might be avoided by adding the diluted molasses containing saccharate of lime to its own bulk of strongly fermenting wash. The carbonic acid of the fermenting liquor would then perform the office of the acetic acid of the dunder, by precipitating the lime as carbonate of lime and liberating the sugar. The idea that dunder is connected with the fine flavour of rum, Horsfield disproved by two experiments. In one trial molasses—from which all the lime was precipitated by sulphurous acid—and water only were used. The resulting rum was a very fine-flavoured spirit, and the yield was perceptibly greater than from molasses not thus treated, though from similar cane. Another trial was made by partially filling the rectifying vessels or retorts, as they are called in Jamaica—they are like the wash-heater, as prepared B, Fig. 45, and connected with the still in the same manner—with dunder, with a view to increase the flavour of the rum, as might be expected if the flavour proceeded from that source. The rum, however, thus obtained had the disagreeable taste of dunder, and was totally different from that of *Old Jamaica*. The flavour of rum appears to depend entirely on the presence of a fusel-oil, the formation of which is immediately dependent on the proportion of the surface of the wash exposed to the air during fermentation, to its entire volume. It is well known that two contiguous sugar plantations will produce very different qualities of rum, though operating in the same identical way. But it has been observed that in such cases the size and the exposed surface of their fermenting vats were different, or if not, the stills were of different capacity. The protracted boiling of the wash in very large and deep stills, injures the flavour by increasing the empyreumatic products. From the above observation it would follow, that the origin of the genuine flavour is intimately connected with the more or less complete oxidation of the molasses ferment induced by the greater or smaller surface of the wash—compared with the bulk—exposed to the air. Large cubical cisterns, holding 1000 and more gallons, yield an inferior rum, as compared with smaller vats having a larger exposed surface. Similar observations made by Liebig with regard to Rhenish wine appear to confirm this opinion.

The second office fulfilled by dunder—an office of some importance for the more rapid development of fermentation—is by its richness in ferment. The colonial distiller does not employ any yeast for inducing fermentation of his wash; he is consequently obliged to work upon more dilute solutions of molasses and skimmings, collectively called *sweets*, than are used by his continental competitors. Any, even the slightest source of ferment, must therefore be welcome for his purpose. Dunder is such a source. Ferments, it is well known, are destroyed by boiling-heat of water, and recent dunder is in that respect perfectly inert; but by exposure to air in shallow tanks an oxidation and regeneration of the *killed* ferment takes place, and it is to this circumstance that a part of the favourable action of dunder must be ascribed.

Rapid fermentation of the wash may be obtained without the assistance of dunder, by converting the cane-sugar contained in molasses into grape-sugar, by treatment with a small quantity of acid, which is subsequently neutralized. Less ferment is required for converting grape-sugar into alcohol than cane-sugar. Some plantations manufacture superior rum though their sugar is nearly as dark as coal-tar. Molasses from such sugar contain chiefly grape-sugar.

Much attention should be paid to proper adjustment of the amount of water employed in the preparation of the liquor. Before commencing, the various boilers and vats are thoroughly washed and freed from saline matter, by hot or cold water.

In the beginning of the distilling season, more sugar is employed than is afterwards found requisite; the reason of this is, that the distiller has no good lees and very little molasses to add to the mass; besides, the scum or froth from the sugar is not so rich from the first boiling of the season as in the months of March, April, and May, which is the most favourable time. The following proportions succeed well at starting:—For every 136 gallons content of the vat, pour in 61 gallons of scum, 7 of molasses, and 68 of water. When the lees and dunder are good, equal quantities of skimmings, lees, and water are employed, and for 100 gallons of this mixture, 10 of molasses are added. Should the pressing-mill be not in operation, and skimmings cannot be obtained, it is found advantageous to employ equal parts of lees and water, and with every 136 gallons of the compound 27 of molasses are mixed. With mixtures such as these, the distiller can obtain from 10 to 15 per cent. of rum and other products; but this quantity depends very much upon the quality of the ingredients operated upon, as also upon the state of the weather and time of distillation; hence, an intelligent distiller varies the proportion of the bodies submitted to fermentation.

Rum differs from what is termed sugar spirit, for it contains more of the natural aroma, or essential oil of the sugar-cane. When the West India distillers have enough of matter, they mix water with it, and allow it to ferment in the ordinary way; the fermentation proceeds slowly at first, on account of the scarcity of the yeast, but as soon as sufficient ferment has been produced, it operates quickly on the whole mass till the attenuation is finished. This liquid is then distilled, and produces a spirit of great strength, nearly equal to alcohol, which they name *double-distilled rum*, or *double rum*. The spirit is more easily concentrated if much liquid be submitted to distillation, but in the course of this operation it yields such a large amount of oily matter, that it cannot be used for a considerable time. For preserving the rum, either for exportation or other purposes, it is found useful to make the double rum so as to form alcohol, or ardent spirit. In this state it occupies only half the volume of the ordinary liquid, and can be diluted with water to suit the taste of the consumer, or to the common strength.

The still for the most part used in those islands, and by which a great saving of fuel is effected, is represented in the annexed engraving, Fig. 41. It consists of two distinct parts: A is the boiler, and B the vessel which contains the fermented wash, to be heated previous to its introduction into the still. The boiler and preparer are placed in such relative positions that the heat of the fire, after doing the required service to A, passes under the vessel, B, and thus communicates heat to the liquid. The plan of the construction is seen in Fig. 42, where the arrows show the course of the flue under both these vessels. The waste heat enters the chimney by a damper-opening at the back. *a* is the passage from the fire under the preparer; *b*, a lid screwed firmly on the vessel, B, which resists the pressure of any vapour generated in this vessel; *c*, a safety valve; *j*, the chimney; *h*, the fire-door; K, the tube which carries off the vapours to the condenser attached to the alembic, which communicates by a pipe not shown, with the preparer, B.

When operations commence, the preparer is filled with the fermented liquor, and likewise the still, to a proper height; the fire is then lighted, and the liquid in the alembic very soon boils, and that contained in the preparer at the same time acquires a temperature approaching ebullition, by the waste heat communicated from the flue beneath this vessel. As soon as the matter in the boiler is exhausted of alcohol, the fire is slackened, the residuary liquid drawn off by the discharge-cock, and the boiler replenished by opening the stopcock of the pipe which connects the alembic and preparer. The fire is again urged, the vessel, B, refilled with fresh liquor, and the distillation proceeded with, as in the previous instance.

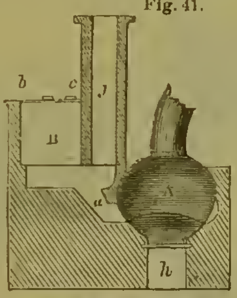


Fig. 41.

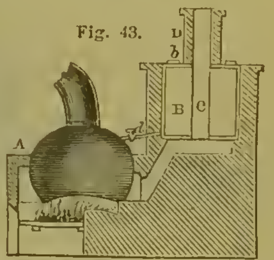


Fig. 43.

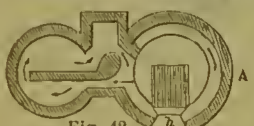


Fig. 42.

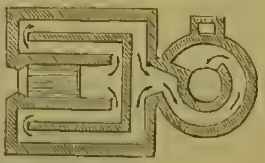


Fig. 44.

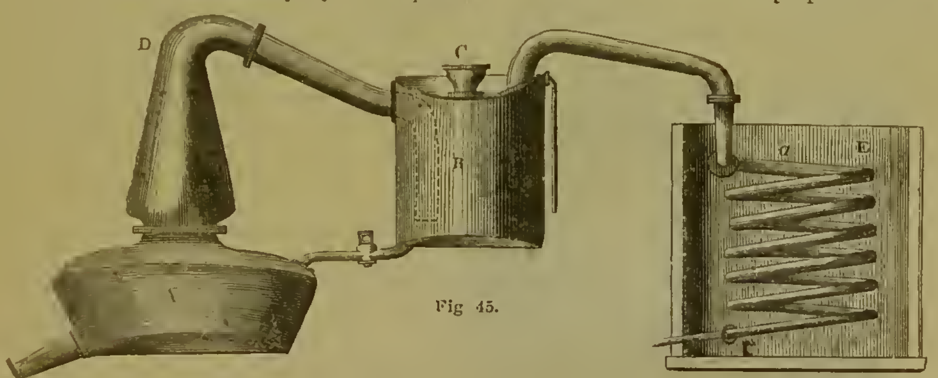


Fig 45.

Another form of apparatus used by the West Indians is seen in the engravings, Figs. 43 and 44. This still and preparer differ only slightly from the preceding. A is the boiler, B the preparer; and the mode of communicating heat to the latter is seen in the plan, Fig. 44, where the course of the heat from the fire is indicated by the arrows, till it enters the circular space of brickwork called the *bonnet;* from this it traverses the perpendicular pipe, C, passing through the middle of the preparer, and thence into the chimney, D. *b* is the lid of the preparer, and *d* a pipe that connects this vessel with the boiler. This arrangement has an advantage over the common furnace, on account of the larger amount of heat it communicates to the boiler flue, and any extra heat is made useful in heating the liquor in the vessel, B.

The annexed form of still, Fig. 45, is also frequently employed in the West Indies. It consists of the boiler, A, with an elongated conical head, D; the preparer, B, into which the wash is introduced by the funnel-basin, C; and the condenser, E, where the spirituous vapour is liquefied in passing through the numerous convolutions of the worm, *a*. The peculiarity of this form of still consists in the communication of the pipe, D, with the preparer, B, by which means the vapour from A is partially condensed in the preparer. The vapour is carried to within a few inches of the bottom of the vessel, B, as shown in the figure by the dotted continuation of D; the liquor in B is heated by the steam condensed in it, as well as by the alcohol which passes through it uncondensed to the worm, *a*. By this arrangement fuel is economized, and a strong spirit is procured at one distillation.

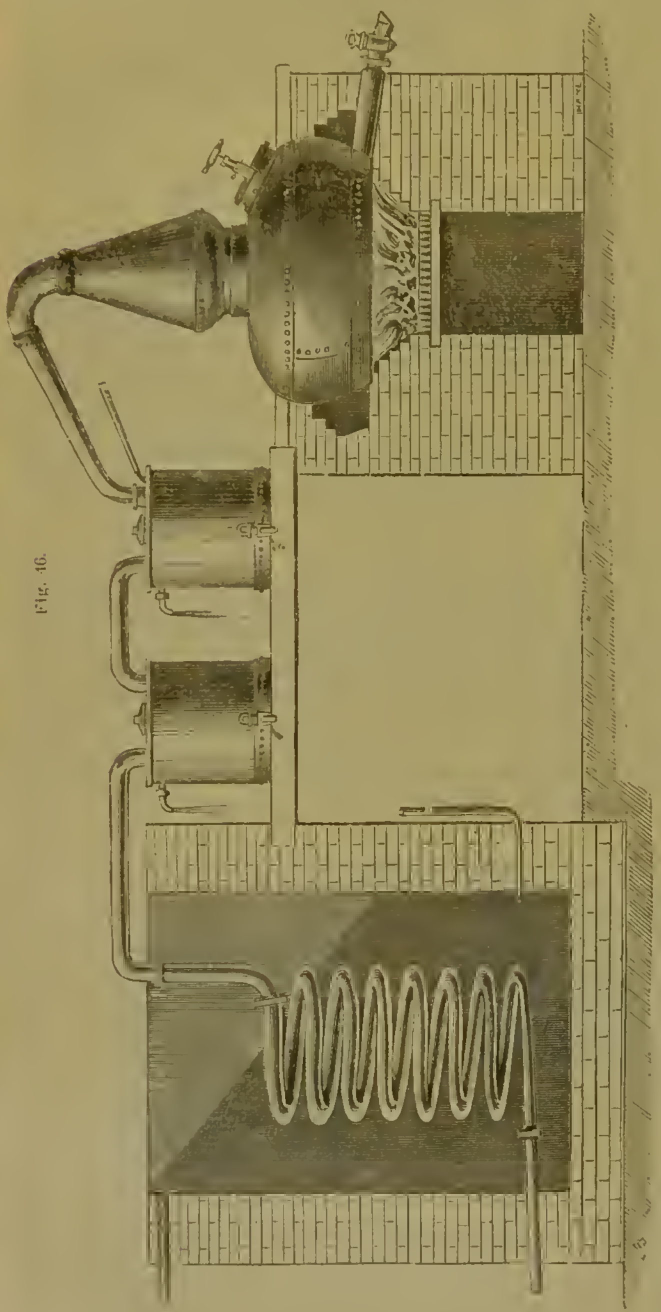


Fig. 46.

Fig. 46 shows Pontifex's still, now generally used for rum in the West Indies and elsewhere. The still is of copper, with a discharge pipe and cock, and a manhole for cleaning, &c. The still is shown set in brickwork and heated by fire, but sometimes it is fitted with an outer casing or jacket of iron, and steam passed between the still and the jacket—the heat necessary for distillation being thus obtained without the direct action of fire. On the top of the still is a conical head of copper, with a neck and arm leading to the next part of the apparatus, which consists of two copper cylindrical vessels, usually called "retorts" by the distiller. Each retort is fitted with a discharge cock and manhole, and in the best construction the top or cover is a few inches below the top of the sides, leaving what is termed a "water chamber." To the retort next the still a small pipe is attached, through which cold water is run to the water chamber, and discharged on the other side, as shown. The same thing is also done with the second retort. The use of these water chambers will presently be seen. The arm pipe that enters the top of the first retort is continued to within a few inches of the bottom. The pipe from the first to second retort is taken from the top of the first, but is continued down nearly to the bottom of the second, in the same manner as the arm pipe in the first retort.

These retorts are on the same principle as Woulfe's bottles (Fig. 7). A pipe is taken from the top of the second retort, and leads to an ordinary condensing worm made of copper or pewter, placed in a tank, frequently made of bricks, through which cold water is kept flowing. The lower end of the worm passes through the tank for discharging the spirit.

The following is a practical description of the method of rum distilling, as conducted in the West Indies, with a still like that just described:—Wash, from which rum is distilled, is composed of sugar skimmings (4 parts), lees of still (5 parts), and molasses (1 part) (the total quantity being the same as the charging capacity of the still to be used), the materials being mixed in a vessel called the "mixing cistern."

The lees of still is a term for the residue left in the still after distillation; and when commencing, of course, no lees are on hand, and then water should be substituted. The wash is pumped into the fermenting vat, and there fermented; the vats are skimmed twice a day during fermentation, which usually occupies fully a week, and when that is completed the fermented wash is ready for the still. The still is charged up to the manhole, and a steady fire kept up. In each retort a quantity of low-wines is put, sufficient to cover the end of the descending arm pipe in first retort, and the pipe from first retort in the second one. In place of low-wines, wash may be used. When, from the heat, the vapour rises from the wash in the still, it passes up the head and down the arm pipe into the first retort: there, the hot vapour coming in contact with the low-wines, a second distillation takes place, and vapour rises to the top of the retort to escape by the pipe leading to the second retort; but the cold water running over the top in the water chamber condenses the weakest portions of the vapour, which fall into the bottom of the retort as low-wines, the stronger vapour only passing into the second retort, where a similar process takes place. The vapour rising from the second retort is freed of its aqueous parts and impurities, and passes into the worm, where it is condensed and discharged as spirit. The spirit thus obtained is much stronger and better than that produced by a still and worm only, without retorts.

The "faints," or low-wines left in the retort after distillation, are drawn off and kept in a cistern until there is enough to make a charge for the still, and then that is worked off in the same manner as fermented wash. Fresh low-wines are used in the retorts at each distillation.

About five hours are required to work off one charge of the still. The charge of wash produces about 10 or 12 per cent. of rum, at an average strength of 25 per cent. over proof.

In some of the islands a still makes about 220 gallons of rum daily; these are produced from about 530 gallons of low wines, or 113 of rum may be procured from 1200 gallons of wash. This liquor is so strong that olive oil will sink in it, and by one rectification it is made to approach the strength of alcohol. The process of distillation is in general slow, and much caution is observed in the condensation of the spirit. To provide against a scarcity of water, which often occurs in the islands, they preserve in large tanks a sufficiency of rain water to enable them to mix the molasses, &c., and to cool the worm of the still. As the water becomes heated in the worm-tub, it is carried to coolers or cisterns, and when cold it serves again for refrigeration. In most of the islands, the curing-houses for sugar and the distilleries for rum are constructed on the sides of canals, and the canes carried to them from the plantation, either in boats or by negroes. Five or six immense copper boilers are kept in each of these houses, and the greatest cleanliness is observed in the distillery; a precaution of immense importance, which must contribute largely to the strength and purity of the rum. In Jamaica the operations go on without intermission; the negroes being formed into divisions or relays, who relieve each other at regular intervals. The richness of flavour peculiar to Jamaica rum has rendered it famous in all parts of the world; this is undoubtedly derived from the raw juice and the fragments of the sugar-cane, which are mashed and fermented with the other materials in the tun. The essential oil of the cane is thus imparted to the wash, and carried over in the distillation. Sugar, when fermented and distilled by itself, yields a spirit in no way different from pure alcohol. Time adds much to the mildness and value of rum; the planters age it fictitiously by the addition of pine-apple juice.

A superior quality of rum is manufactured in the colony of Demerara, where distillation has been carried to a high state of perfection by the perseverance and skill of several scientific men, who have caused the rum of this district, and that of Essequibo, to be as much prized in the American market as Jamaica rum is in England. In Brazil large quantities of rum are manufactured, which are exported to America and to most European nations. The process followed is rude and simple. The wash is generally fermented in large earthen jars, but no fixed rules to regulate the quantity of molasses which should be operated upon are observed. A strong lie is poured on the syrup, in order to thicken and purify it, which is obtained by burning a plant of the *polygonum* species, called by the Indians *cataya*, and infusing the ashes in water. This plant has a bitter pungent taste, and is considered of use in making rum. The stills are mere earthen jars, with a long narrow neck, on the top of which is placed a head or cap, having at one side a pipe of about 6 inches long; to this adapter a copper tube, 4 feet in length, is connected, which passes through an earthen vessel sufficiently large to hold the water for the condensation of the spirit, and this contrivance is made to answer the purpose of both worm and worm-tub.

To calculate the cost of rum to the sugar planter is difficult; in general, it is estimated that one-fourth of the entire produce of a plantation may, in point of value, consist of rum, and accordingly one-fourth of the expenditure may be taken as the first cost of the rum, and the remaining three-fourths as that of the sugar. Some say that the charge of making rum bears a similar proportion to that of home-made spirits, but this is an erroneous assumption. Rum is made from the molasses, or that part of the cane-juice which will not crystallize into sugar, and also from the scum which is taken off during the saccharific process, and which in sweetness is equal to one-fifth of molasses. Take, as a standard, a distillery on a plantation producing 250 hogsheads of sugar, yielding 15,000 gallons of molasses, and scum equal to 5000 gallons, netting in all 20,000 gallons of molasses. These would produce about 15,000 gallons of proof rum, which, when brought to the British market, would be reduced by the voyage to about 13,500 gallons, the average loss being 10 per cent. These would cost the manufacturer throughout the islands from 1*s.* 1*d.* to 1*s.* 4*d.* per gallon, independent of all charges for puncheons, freight, commission, and other unavoidable expenses.

From this statement it appears that the distiller of rum has little or no profit, but being the grower of the material, and having his capital embarked in the trade, he is compelled to manufacture it from necessity, and the sooner he can turn the article to account the better he is enabled to bear loss and meet his engagements.

In France a large quantity of spirit is annually manufactured from the molasses of the beet-root sugar factories, of which a great many exist in that country. The better sort of molasses remaining after the refining of the sugar of the colonies is too valuable to be converted into rum, but sometimes the inferior article is disposed of in this way. On the arrival of the molasses at the distillery, they are emptied into large cisterns perfectly free from any dampness which would cause them to ferment, and here they remain till required for use. The fermentation of molasses presents some difficulties; they must be properly mixed with water in such proportion that the resulting liquid will not exceed 8° of BEAUMÉ's areometer, at a temperature of 20° C. = 68° Fahr., which should always be the heat of the mixture. When too little water is used the fermentation sets in too rapidly, the temperature becomes higher, and acetous fermentation speedily ensues. On the contrary, when too much water is employed the fermentation is inactive; in consequence of the low temperature a longer time is required, and generally bad results follow. These inconveniences can be overcome by attention to the directions about to be given.

It sometimes happens that the fermentation of the saccharine solution suddenly ceases, and cannot be revived by an increase of temperature or addition of a stronger solution of molasses. This is owing to the presence of lime and potassa, which are contained in almost all the molasses of beet-root sugar, and in consequence of the alkaline reaction imparted by these substances to the liquid, the conversion of the sugar into alcohol is interrupted. This anti-fermenting property of alkaline bodies is very easily removed; it suffices to add a certain quantity of sulphuric acid to neutralize them and form sulphates, in which state they are inert. A slight excess of acid might be employed without prejudice to the proper degree of attenuation, or to the taste of the product; for the molasses always contain salts of organic acids, with potassium, &c., which are decomposed by the sulphuric acid, and the organic acids are liberated. The sulphuric acid is to be added when the water is mixed with the molasses, and may vary in amount from half a per cent. of the weight of the latter, as a minimum, to 1½ per cent. as the maximum quantity. The molasses being comminuted with the water and acid, so that the solution stands at 8° of the areometer, about 2 per cent. of their weight of fresh barm, pressed and previously diluted with water, is added; the liquid is then strongly agitated and left to ferment. The fermentation is made in a number of tuns whose size corresponds with that of the distilling apparatus, and by this arrangement the distiller is enabled, when the fermentation in one tun is finished, to distil the contents directly.

The fermented wash should never remain longer than twenty-four hours before it is distilled. From this it is manifest that the distiller should be furnished with as many fermenting vessels as will permit him to have the contents of one tun daily ready for distillation, and one ready for charging each day; the intermediate tuns being in a higher state of fermentation as their turn brings them nearer the proper time of their being distilled. All the tuns should be well covered, to prevent the contact of the atmosphere and the acid fermentation taking place.

If molasses be fermented and distilled merely for the alcohol which they yield, the preceding directions relative to the proper dilution and strength upon the areometer answer best; some distillers, however, in addition to the alcohol, extract the alkaline salts; and in this case the preceding strength of 8° on the areometer would offer an inconvenience, inasmuch as a large amount of fuel would be consumed in evaporating the residuary liquid after the alcohol was expelled. To prevent such expenses for combustibles, an investigation was instituted to find out a means for fermenting the saccharine wash at a greater density than 8°, and the endeavour proved successful. The high density is 14° of the areometer, and in order to ferment such a solution without its passing to the acid fermentation the following mode is adopted:—When a sweet wash of 14° B. is set to ferment, the temperature of the liquid rises to 86° Fahr. (30° C.) in twenty-four hours, at which degree the alcohol is readily transformed into acetic acid. To oppose the formation of acetic acid, from the high temperature already mentioned, it is necessary, as soon as the liquid marks 76·6° Fahr. or 27° C., to divide it into two equal portions, and add to each half as much molasses of 14° strength as it already contains. Previous to mixing the second portion of molasses with the fermenting liquor, they should be well agitated with 2 per cent. of their weight of barm. Fermentation is now allowed to proceed without apprehension of the temperature rising so high as to favour the acetous fermentation in the liquor.

M. LAUGIER's apparatus, which will be subsequently described, is expressly adapted for the distillation of fermented saccharine liquors, though wine and malt wash are also distilled in it. It works upon the same principle as DEROSNE's still, under a simpler construction. In France, where considerable quantities of molasses are converted into rum, and this apparatus is in operation, the distillery is divided into the store-room, where the stock of molasses is retained until required for use; the fermentation-room, the still-room, and the store-room for the finished spirit.

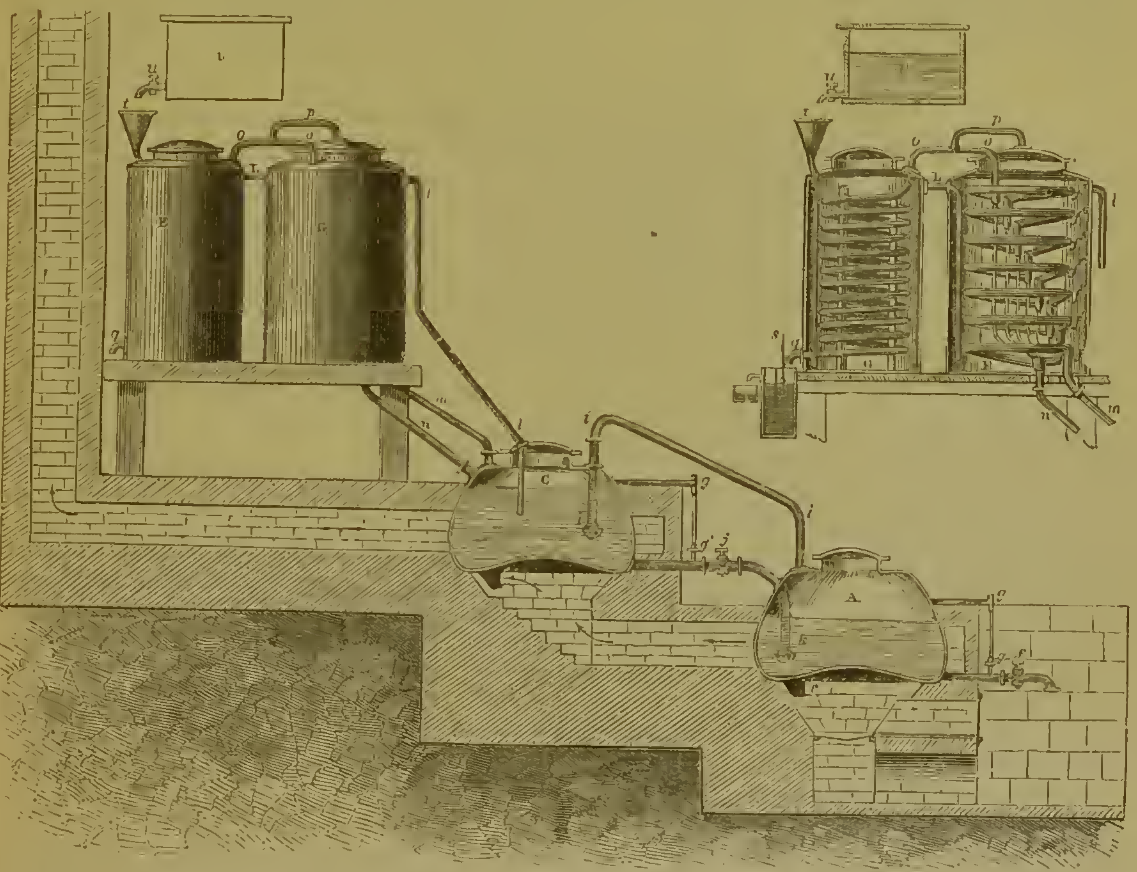
The fermenting tuns are of a size to correspond with the quantity of wash which the still is capable of working daily, and these tuns are worked in rotation, so that one may be worked off and ready for distillation each day; by this means the fermented liquor is prevented from being exposed to the air, and the formation of acetic acid wholly prevented.

A side view of the apparatus, as it appears set in brickwork, is seen in Fig. 47; A and C are sectional views of the boilers heated by the fire, *c*, under A, round which the flue passes; thence in the direction of the arrows round the second boiler, C, in a similar way, and afterwards into the chimney; G is the rectifying cylindrical vessel, and E the refrigerator where the spirit is condensed. The boiler, A, is furnished with two pipes; one of these is for discharging the contents when all the alcohol is expelled, and is furnished with a stopcock, *f*; the other pipe, *i i i*, carries off the generated vapour to the next boiler, C, where it terminates in a perforated rose, within a short distance of the bottom of the vessel.

A pipe issuing from the bottom of C, and furnished with a stopcock, *j*, enters the first boiler and terminates, like the pipe, *i*, in a perforated rose, as seen at *k*. From C, the pipes, *m*, *n*, and *l*, rise; *m* and *n* are connected with the rectifying apparatus in the cylindrical vessel, G; the former carries off the vapours generated in C, and the latter returns the liquid condensed in this vessel into the boiler. The pipe, *l*, serves to charge the boiler, C, from the rectifying apparatus. Two pipes, *o o* and L, unite the rectifier and the condenser; the former conducts the uncondensed vapours from the rectifier to the worm in the refrigerator, and the latter serves to replenish the vessel, G, with liquor from the refrigerator. The pipe, L, descends to within a short distance of the bottom of G, so as to have the colder liquid issuing in contact with the pipes which contain the warmer vapours. Another pipe, *p*, emerging from the cover of the rectifying vessel, conducts any vapour generated by the condensation in part of the distilled products of the boilers, A and C, to the condenser. The refrigerator is filled through the funnel tube, *t*, from the tank, *v*, by means of the pipe and stopcock, *u*.

Fig. 47.

Fig. 48.



The manner of working the still is simple:—Liquor is allowed to enter the funnel tube, *t*, until it begins to flow down the pipe, *l l*, into the boiler, C; and as soon as this is observed the stopcock, *j*, is opened, and the liquor admitted into the first boiler until it rises a few inches above the rose, *k*, as shown by the glass gauge, *g g*; the stopcock, *j*, is shut, and the liquor allowed to flow into C until it rises above the end of the pipe, *l l*, which emerges into the liquor; this is shown by another gauge pipe, *g′ g′*, attached to C. The stopcock, *u*, is now closed, and the fire urged under the first boiler, the contents of which very soon boil, and are partly converted into vapour, which is emitted to the next boiler through the pipe, *i i*. By means of the heat abstracted from the vapour in passing through the liquid, and that communicated to the boiler by the flue which circulates round C, the contents of this vessel are also made to boil, and, like those of A, are partly vaporized; the vapour is forced through the pipe, *m*, into the rectifying vessel, where, by means of a peculiar arrangement of pipes—to be explained further on—the greater part of the steam which is forced along from A and C is condensed, and falls back into the boilers through the pipe, *n*, to undergo a second distillation. The first portions of the vapour that enter this vessel are entirely condensed, in consequence of the liquid contained in it being as yet cold; but after a short time the heat of the condensing vapour raises the temperature of this liquid so high that it will not condense any longer the richer alcoholic vapour, and the latter rises through the pipe, *o o*, and enters the worm in the vessel, E, where complete condensation takes place, and the spirit produced flows out by the pipe, *q*. As soon as the liquor in the first boiler becomes exhausted the fire is slackened for a short time, the contents discharged through the pipe and stopcock, *f*, and a further quantity admitted by the pipe and stopcock, *j*, from the boiler, C, and this again replenished by opening the stopcock, *u*, of the tank, *v*. The fire is again urged briskly, and thus the distillation proceeds successively, so as not to lose alcohol.

Fig. 48 is a section of the rectifier and refrigerator: in the first there are seven compartments formed of large circular pipes, as seen at $f\ f'\ f''\ f'''\ f^5\ f^6\ f^7$, each one of which terminates in a smaller pipe that meets the others in a ball at the end of the pipe, *n*. *o o* shows the connection of this apparatus with the refrigerating worm in the vessel, G; *p*, the pipe connected with *o o*, and emerging from the cover of the vessel, E, for the purpose of carrying off any vapours generating in the liquid surrounding the apparatus in this vessel. *t* is the funnel pipe, reaching to the bottom of the refrigerator; and *q* the termination of the worm, by which the finished spirit is discharged into a small covered vessel, *r*, which contains an alcoholometer, *s*, to indicate the strength of the spirit.

The fermenting molasses are so viscous, when they are treated in such a way as to mark only 8° of the areometer, that at some period the mixture intumesces, so as to overflow the fermenting tuns, unless the latter be very large. To obviate this, some soft soap is added, which, being partly decomposed by the slight excess of acid contained in the liquid, the oily portion forms a layer which destroys the homogeneousness of the syrupy effervescence, and disposes the bubbles of carbonic acid to burst and pass off without much rising in the liquid. Fermentation is known to be finished when the action, after having been regularly increasing, ceases suddenly, and the temperature subsides. A sign by which a good fermentation is distinguished is the falling in of the head, and the diminution of specific gravity from 8° to 1° Beaumé.

If the liquid, after attenuation, be not immediately distilled, it is necessary to cool it down rapidly, to prevent its being converted into acetic acid. This is done either by passing cold water through a coiled pipe in the fermenting tun, or by emptying the whole into sulphured tubs, which resist the further action of any fermentation. Of the two methods the worm is preferable, as in a slow fermentation the transmission of hot water through this pipe would revive it; and when the temperature of the liquid becomes higher than what it should be, from a too rapid fermentation, it is equally opposed by pouring cold water through the worm. LAUGIER'S apparatus is best adapted for the distillation of the fermented liquor of molasses.

The quantity of spirit obtained from molasses, when fermented at 14°, is less than when the mash is made to mark 8°; but considering the advantage of obtaining the alkaline matter, there is less evaporation, and consequently less fuel consumed. A method has been lately introduced by DUBRUNFAUT, by which the attenuation is made at 8°, and the saline matter obtained without much extra fuel. One part of this process is to employ the spent liquor, after distillation, for bringing a second portion of molasses to 8° of the areometer; this liquor has no injurious effect upon the fermentation, and offers the advantage of having double the quantity of salts in the same bulk of liquid.

This liquid he introduced into a steam boiler instead of water, for the purpose of generating steam to heat the liquid for distillation, and the apparatus. Finally, the evaporation is finished in three pans or boilers, which are partly heated by the waste heat from a reverberatory furnace. The degree of heat which the liquid has, before it is run into the furnace, is 32°: 1000 kilogrammes of molasses afford from 100 to 140 kilogrammes of saline matter, marking 50° to 55° on the alkalimeter.

POTATO SPIRIT.—Potatoes afford a considerable quantity of alcohol; and of late years the manufacture has been extensively conducted in France. There are two methods practised. In the first the starch of the potato is fermented without any previous preparation, and in the second the starch is converted into sugar by sulphuric acid. If a quantity of starch, no matter whether obtained from wheat or potatoes, be incessantly boiled with water acidulated with sulphuric acid for a few hours, occasionally adding water as it evaporates to preserve perfect fluidity; then saturated with lime, boiled to separate the sulphate of lime; and lastly, the solution be concentrated; a dark syrupy liquid is obtained, which on cooling affords abundance of sugar in crystals. This sugar certainly differs in some respects from common sugar; it is not quite so sweet, nor so soluble in water; it crystallizes differently, fuses at a much lower heat, and its solution ferments with great facility. It has been found that, during the whole process of its formation, not a bubble of gas is discharged, that the sulphuric acid remains unchanged, and that the contact of air is unnecessary.

There is produced from 100 parts of starch about 110 parts of sugar, which is converted by fermentation into alcohol. The advantages afforded by converting potatoes to this use are, that they are cheap and afford a good spirit, while the residuum of the distillation is good food for cattle; grain is economized and less yeast is consumed.

To obtain spirit according to the first process the potatoes are to be well steamed for an hour, and then bruised between two cylinders of wood or sandstone. Ground malt is mixed in a keeve with warm water, in quantity sufficient to give the consistency of thin pap, and the potato paste is then added, the whole being well stirred with a proper quantity of water until no lumps remain. The stirring is to be renewed at intervals until the mixture is cold. Natural yeast of beer, or that made artificially from rye, is introduced, but in less quantity than would be required for corn, since potatoes ferment more easily. Experience has proved that the addition of red beetroot or carrots to the potatoes and malt affords spirit of a better flavour and in larger quantity. When the fermentation has been pushed to its utmost the whole matter, both liquor and sediment, is introduced into the still, and distilled as in any other case, proper precautions being taken to prevent burning.

The process recommended by SIEMEN, and now applied in Denmark, is to heat 3 or 4 tons of potatoes in steam a little above 212° Fahr. (100° C.), then to mash them well by the rotatory motion of an iron cross in the same vessel wherein they are steamed, and to add hot water, alkalized with 1¼ lbs. of caustic potassa. All the mucilage, which in the boiled potatoes commonly remains insoluble, is by the addition of the alkali converted into a starch, which easily passes through the sieve, leaving the thin skin of the potato. The water is to be in such quantity as to make a thin paste; this being quickly cooled, yeast is added, and the process conducted in the usual manner. It is said that by this method the quantity of spirit from a given weight of potatoes is greatly increased: 50 hectolitres, 30 litres (137·64 imperial bushels) of potatoes, along with 8 hectolitres (22·12 imperial bushels) of ground malt, yield 9 hectolitres (198 imperial gallons) of spirit. CADET states that 800 lbs. of potatoes will afford 30 lbs. of spirit, which at that time he calculated to cost the distiller 36 francs, and to sell for 48.

The process for procuring alcohol from potato sugar need not be particularly described. The sugar being once obtained from potato starch, it is easy to conduct the fermentation. During the conversion of the starch into sugar, a few drops of a solution of iodine in alcohol is frequently added to a small portion of the liquor, to see if the blue iodide of starch is formed; this reaction manifests itself as long as any undecomposed starch remains. From 50 kilogrammes (110·31 lbs. avoirdupois) of potato starch, converted into sugar by sulphuric acid, are obtained from 20 to 25 litres (4·4 to 5·5 imperial gallons) of alcohol, at 0·935.

According to WUNRICH, starch requires but 1 or 2 per cent. of sulphuric acid to convert it into sugar, if the heat applied be a few degrees above 212° Fahr. (100° C.), and two or three hours are then sufficient to crystallize it. He applies steam heat in wooden vessels.

The yeast thrown up by potatoes during fermentation, even with one-fifth of their weight of barley, possesses but little energy, and is therefore not used in attenuating the potato wash.

In the experiments made under the personal inspection of Professor OERSTED at Copenhagen, from 16½ to 17 quarts of spirit, at 50° of Tralles' alcholometer, were obtained from a ton of potatoes, making a fair allowance for that portion of the product due to the malt used in the maceration. This spirit had a good flavour, though the produce was inferior to that obtained by the French chemists. MULLER asserts that an apparatus on SIEMEN'S principle (Figs. 35, 36), the expense of which is about 250 Prussian dollars, is capable of producing 50 per cent. more spirit from potatoes than the apparatus generally used in Germany.

About the year 1832 Messrs. CALDER, at Eyemouth in Berwickshire, distilled spirit from potatoes for some little time. The spirit, which had the flavour of hollands, was pure and good, and it was affirmed that no grain or malt was used in its preparation. The fermentation was described as beautiful, the head rising 7 or 8 feet like clouds of cotton; and when beaten down to the surface of the worts it rose again in the same majestic manner. The gravity worked at was 40°, and the attenuation was good. The potatoes were ground in a mill like a common pepper mill in shape, but made of sheet iron perforated like a grater. The pulp thus produced was mashed in the keeve with boiling water, and the extract ran off quite pure and freely. A sperge, or small worts of about 20° gravity, was obliged to be used, otherwise the worts at the noticed gravity of 40° could not be got off; the produce was good, as there was no deficiency. The spirit sent to the London market, when called grain spirit in the permits, was highly prized; when this error was corrected, and the product was denominated spirit distilled from potatoes, the price fell, and it was not so much in vogue. About the same time JAMIESON of Fairfield, near Enniscorthy, commenced distilling from potatoes. They were sliced, dried on a cornkiln, ground to flour, mixed in certain proportions with grain, and mashed in the ordinary manner. But the manufacture was abandoned in consequence of the opposition of the peasantry, through fear of a scarcity of the article of food.

From some late experiments, DUBRUNFAUT proposed to brew from the starch of potatoes an excellent beverage resembling French beer, the starch being macerated and fermented with hops. By fermenting the saccharized starch with honey instead of hops, a palatable liquor was made, having all the qualities of Louvaine beer. Potato starch, being free from any peculiar taste, seems capable of receiving flavour in its fermentation from any of those substances which are used to give their peculiar characteristic tastes to the various kinds of beer and home made wines.

HARE, having observed a strong analogy between the saccharine matter of the sweet potato and molasses, or the saccharum of malt, boiled a wort made from the potatoes, of 1060° specific gravity, with a proportionate quantity of hops for the space of two hours. It was then cooled to about 56° Fahr. (17°·7 C.), and yeast added. As far as could be judged, the phenomena of the fermentation and the liquor produced were precisely the same as if malt had been used. The wort was kept in a warm place until the temperature was 85° Fahr. (29°·4 C.), and the fall of the head showed the attenuation to be sufficient; yeast subsequently rose, which was removed by skimming. A further quantity of yeast was precipitated by refrigeration, from which the liquor being decanted, became tolerably fine for new beer, and resembled in flavour ale made from malt. It has been computed that 5 bushels of potatoes would produce as much wort as 3 bushels of malt, while the residue, as food for cattle, would be worth half as much as the potatoes.

In the opinion of some—particularly those who have not employed sulphuric acid in saccharifying the starch—the best time to use potatoes in distillation is in spring, when they begin to vegetate. The growth of the buds must be checked, as in the process of malting; and this is easily done by spreading them on a floor, and by subsequent turning, so as to deprive them of as much of their water as possible. When reduced to a pulpy consistence, diluted with boiling water, and drawn off and cooled to a proper temperature, the liquid is then fermented in the same manner as grain worts; and such has been the treatment observed by many who have tried the distillation of potatoes in this country. Sprouted potatoes produce as perfect farina in July as in December, and equal in quantity to what they would have yielded earlier in the season, being, according to Sir John Sinclair, about 14 lbs. per cwt.

It has been stated that potato apples give, by proper treatment, as much alcohol as an equal quantity of grapes, when bruised and fermented with one-eighteenth or one-twentieth of their weight of yeast.

From these details on the application of potatoes in the manufacture of spirit, persons may be induced to try experiments that might ultimately prove advantageous. If they proceed by reduction of the farina to a pulpy substance, the operation is simply by boiling; if by the production of starch, it may be mechanically effected at little expense and labour, either by pounding or grating, and elutriation with cold water.

In some parts of France the tuber of the Jerusalem artichoke—*Helianthus tuberosus*—has been used for the purpose of distillation. The wash extracted from this vegetable, when fermented in the ordinary way, is found to yield a very pure and strong spirit, which is said to resemble that obtained from the grape more than any other substance that has hitherto been tried.

The root grows luxuriantly almost in every climate, but it does not appear that it has been cultivated much in England, either for the production of spirit or other uses; it might be remunerative in this particular, in producing a medium beverage between genuine French brandy and the fiery spirit extracted from grain, and sold as gin and whisky.

Arrack, contracted into rack, is a spirituous liquor from the East Indies. The name is used in the East to signify any alcoholic liquor; but that usually bearing this name is a liquid distilled from *toddy*, the juice of the cocoa-nut tree, *cocos nucifera*, and procured by incision. In all countries where rice is abundant, an alcoholic liquor is distilled from it, called *arrack* or *rack*. Goa and Columbo arrack are always made from toddy; Batavia and Jamaica arrack from molasses and rice, with a little toddy. The Pariah arrack contains *cannabis sativa* and a species of *Datura*, which render it more inebriating; it is not, however, certain whether the Pariah arrack is used generally to imply a sophisticated spirit, or is only applicable to that liquor with which the above ingredients have been compounded.

The process is nearly the same as that for making grain spirit. Rice is put into a vat, covered with water, and agitated. A handful is from time to time taken from different parts of the vat, and germination is allowed to proceed until at least half of it has sprung. The operation may be hastened by adding lukewarm water, and drawing a certain quantity from the top, heating and returning it to the vat, the contents of which are well stirred. Great caution is necessary in doing this, for much risk is run of breaking the seed, which would make the rice decay, and hinder the fermentation of the rest. If such a thing occurs, the injured grain might be extracted, but this would be attended with so much labour, as compared to the value of the rice, that commonly the whole is rejected by distillers, and sold for the use of cattle. To avoid these mishaps, a man accustomed to the work is employed. He introduces the rake just below the surface of the rice, agitates the water in turning, and stirs gently till he reaches the bottom; the same caution is observed in bringing the rake again to the surface. When fully half of the rice is germinated, the plug at the under part of the vat is withdrawn to let out the water; the rice is then removed to a room, and heated like the barley in the distillation of the grain spirit. It is submitted to a heat of 59° Fahr. (15° C.), which finishes the germination.

The subsequent operations are the same as those pursued by the brewer. When the rice has sufficiently acquired the vinous fermentation, it is introduced into the still, and treated like the other substances discussed.

In India, when the material for distilling, whether rice or the simple fermented juice of the *cocos nucifera*, is ready, a hole is dug in the earth, suited to the size of the still to be used. On a level with the bottom of this hole there is an underground communication made for the purpose of feeding the fire with atmospheric air; near the edge of this orifice is a chimney, serving both for the supply of the fuel and for the escape of the smoke; a fire of dry wood is first lit, and when the ground is completely heated, the still is fixed in it, and so bound round with earth as to prevent the escape of any heat. When ebullition commences, and the steam begins to ascend, an

Indian pours a gentle stream of water either upon the head of the still or on the broad and thin surface of a plate of tin or copper, with a gutter for the water to run off, which is fixed on a pan, with a hole in the bottom, and luted to the neck of the still to serve as a condenser. The extreme cold produced by the evaporation of the water on so broad a surface, occasions the vapour from the still to be immediately condensed, and to flow in a trickling stream into the receiver.

A lady who resided in India thus describes the working of a native still which she had an opportunity of seeing :—The still was simply constructed : round a hole in the earth a ledge of clay four inches high was raised, with an opening half a foot in width for the purpose of supplying fuel. Upon the clay a large earthen pot was luted ; to its mouth was closed with lute the mouth of a second pot ; and where they joined, an earthen spout a few inches long was inserted, which served to let off the spirit condensed in the upper jar, the latter being kept cool by a person pouring water constantly upon it. In the cottage, or still-house, was a woman employed in cooling the still by pouring water on it from a cocoa-nut ladle. The woman said that she sat at her occupation from sunrise to sunset, without scarcely a change of position, while her husband constantly brought toddy for distillation.

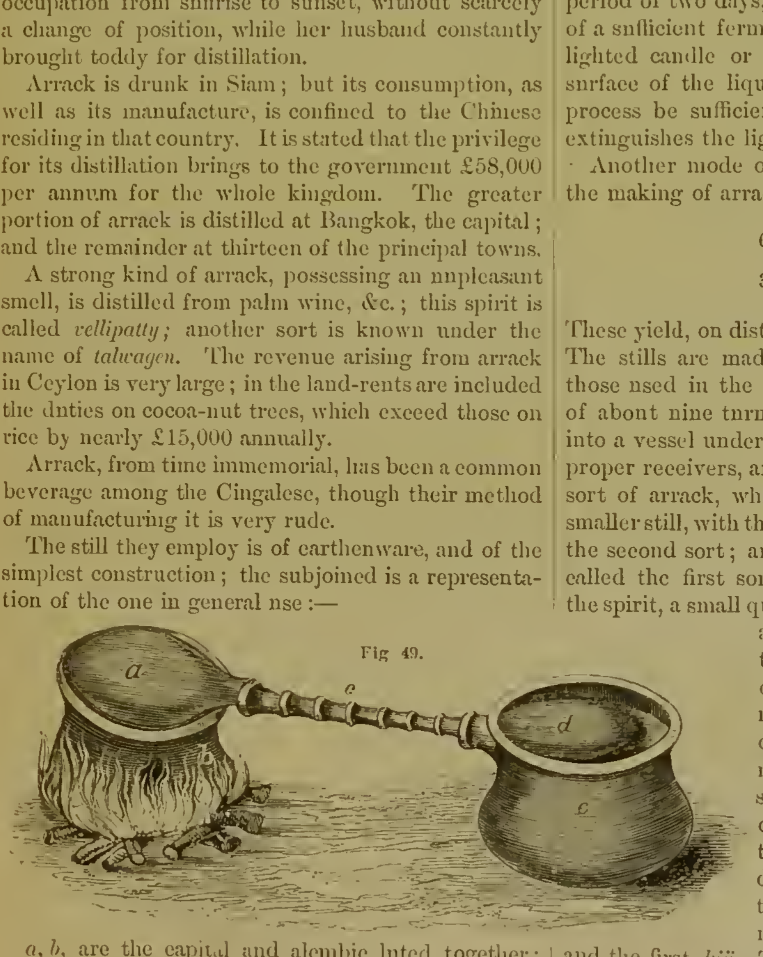
Arrack is drunk in Siam ; but its consumption, as well as its manufacture, is confined to the Chinese residing in that country. It is stated that the privilege for its distillation brings to the government £58,000 per annum for the whole kingdom. The greater portion of arrack is distilled at Bangkok, the capital ; and the remainder at thirteen of the principal towns.

A strong kind of arrack, possessing an unpleasant smell, is distilled from palm wine, &c. ; this spirit is called *vellipatty;* another sort is known under the name of *talwagen.* The revenue arising from arrack in Ceylon is very large ; in the land-rents are included the duties on cocoa-nut trees, which exceed those on rice by nearly £15,000 annually.

Arrack, from time immemorial, has been a common beverage among the Cingalese, though their method of manufacturing it is very rude.

The still they employ is of earthenware, and of the simplest construction ; the subjoined is a representation of the one in general use :—

Fig 49.



a, *b*, are the capital and alembic luted together ; *d*, *e*, a receiver and a refrigerator, in one piece, the former connected with the head by a bamboo, *c*.

Ardent spirit is manufactured in much larger quantities in Java than in any other island in the Indian ocean, which may, no doubt, be accounted for by the great industry of the Dutch, and the celebrity which the Batavian arrack so early acquired under their patronage.

It is made in the following manner :—About 70 lbs. of *ketan*, or glutinous rice, are heaped up in a small vat ; round this heap 100 cans of water are poured, and on the top 20 cans of molasses ; after remaining two days in this vat, the ingredients are removed to a larger vat adjoining, when they receive the addition of 400 cans of water and 100 of molasses. Thus far the process is carried on in the open air. In a separate vat within doors, 40 measures of palm wine, or toddy, are immediately mixed with 900 of water and 150 of molasses, both preparations being allowed to remain in this state during two days. The first of these preparations is carried to a still larger vat within doors ; and the latter, being contained in one placed above, is poured upon it through a hole bored for the purpose near the bottom. In this state the entire preparation is allowed to ferment for two days, when it is poured into small earthen jars, containing about 20 cans each, in which it remains for the further period of two days, and is then distilled. The proof of a sufficient fermentation is obtained by placing a lighted candle or taper about 6 inches above the surface of the liquor in the fermenting vat ; if the process be sufficiently advanced, the carbonic acid extinguishes the light.

Another mode of apportioning the materials for the making of arrack is—

62 parts molasses,
3 do. toddy,
35 do. rice.

These yield, on distillation, 23½ parts of proof arrack. The stills are made of copper, and are much like those used in the West Indies ; the worms consist of about nine turns of Banca tin. The spirit runs into a vessel under ground, whence it is poured into proper receivers, and is called the third, or common sort of arrack, which by a second distillation in a smaller still, with the addition of some water, becomes the second sort ; and by a third operation, is what is called the first sort. To ascertain the strength of the spirit, a small quantity of it is burned in a saucer, and the liquid remaining measured; the difference between the original quantity and the residue gives the measure of the alcohol lost. The completion of the first sort does not require more than ten days, six hours being sufficient for the original preparation to pass through the first still. The Chinese residents, who conduct the whole of this process, call the third, or common sort, *sichew ;* the second, *tanpo ;* and the first, *kiji.* The latter two are distinguished as arrack *api.* When cooled, it is poured into large vats in the storehouses, where it remains until put into casks.

The making of arrack is distinct from that of sugar, which is manufactured to a great extent in Java. The arrack distillers purchase the molasses from the sugar manufacturers.

A very large quantity of arrack is consumed in the East; 700,000 gallons are annually exported from Ceylon, of which upwards of 30,000 come to England.

Carrot Spirit.—In the Transactions of the Royal Society of Edinburgh is a paper by Hunter and Hornby, in which they give the details of the process for the production of the above spirit. With the paper was sent a sample of the spirit, and the society appointed Black, Hutton, and James Russell to investigate this account, together with the specimen of the spirit, and to report upon the same, which they did as follows:—

The sample of spirit which was sent by Dr. Hunter, of York, to the Royal Society, has been examined, and also the account of the experiment on the fermentation and distillation of carrots, by which the spirit was produced. The experiment was made by Mr. Thomas Hornby, druggist in York, with 1 ton and 8 stones of carrots, which, after being exposed to the air a few days to desiccate, weighed 160 stones, and measured 42 bushels; they were affused with water, topped and tailed, by which they lost in weight 11 stones, and in measure 7 bushels; and being then cut, were boiled with the proportion of 24 gallons of water to one-third of the above-mentioned quantity of carrots, until the whole was reduced to a tender pulp, which was done by three hours' boiling. From this pulp the juice was readily extracted by means of a press, and 200 gallons of juice were produced from the whole quantity.

The juice was reboiled with 1 lb. of hops for five hours, and then cooled to 66° Fahr. (18°·8 C.), and 6 quarts of yeast being added, it was set to ferment. The strong fermentation lasted forty-eight hours, during which time the heat abated to 58° Fahr. (14°·4 C.); 12 gallons of unfermented juice, which had been reserved, were then heated and added to the liquor, the heat of which was thus raised again to 66° Fahr. (18°·8 C.), and the fermentation was renewed for twenty-four hours more, the air of the brew-house being all this time at 44° and 46° Fahr. (6°·6 and 7°·7 C.) The liquor was now turned, and continued to work three days from the bung; lastly, it was distilled, and the first distillation was rectified next day without any addition. The produce was 12 gallons of spirits. It resembled the best corn-spirit in flavour, and was proof. The refuse of the carrots weighed 48 stones, which, added to the tops and tails, made provision for hogs, besides the wash from the still, which measured 114 gallons.

From the above, Dr. Hunter draws the annexed comparison between the distillation of carrots and grain:—

Twenty tons of carrots will make 200 gallons of proof spirit. Eight quarters of malt, or rather materials for distillation, consisting of malt, wheat, and rye, will also yield 200 gallons of proof spirit. The refuse from the carrots will be 960 stones, which, at one penny per stone, will sell for £4. The refuse or grains from the malt, &c., will be 64 bushels, each bushel about 3 stones, which, at one penny per stone, will sell for 16*s*. The doctor, however, supposes that the manufacture of the spirit from carrots may be attended with more expense than that from malt, but imagines that the greater value of the refuse may compensate for that expense, and that the saving of corn for other purposes is an object worthy of attention and of encouragement.

Milk Spirit.—The Tartars and Kalmouks prepare a spirit from the milk of mares or cows, which they greatly relish: 21 lbs. of milk yield 1½ ounces of an insipid distillate, and 40 ounces of spirituous liquor; the latter, when rectified, gives 6 ounces of alcohol. Oseretskowsky says that skimmed milk, when deprived of its butyraceous portions, neither produces spirit *per se*, nor by the addition of a ferment. Secondly, milk retaining a portion of its cream, agitated until it commences fermentation, produces alcohol, but in small quantity. Thirdly, the entire milk kept in a close vessel, which by agitation commences fermenting, furnishes more spirit. Nearly the same amount of spirit is procured from the same milk, when a ferment is added to it. Fourthly, milk deprived of the most part of its caseine furnishes very little spirit. Fifthly, when the serous part only of the milk is distilled, it affords little spirit. Sixthly, milk which is fermented in a close vessel, and left for some time, loses its acid, and furnishes much more spirit than it would do if distilled immediately. Seventhly, if the heat of the fermented milk be sustained, the alcoholic portion passes into vinegar.

Madder Spirit.—Within the last few years a patent has been taken out in France by Jullien, for distilling a spirit from the washings of madder, which were previously allowed to run waste. Several madder distilleries are now established in France, and one has been lately erected in Glasgow by Mr. J. Hinshaw, of the firm of Messrs. Arthur and Hinshaw.

To explain the economy of this remarkable process, it may be stated that the madder is imported into this country in the form of a root, which somewhat resembles liquorice, or the stem of heather. To prepare it for the dyer, and especially for dyeing the celebrated turkey red, which has long been a staple business in Glasgow, the root is first roasted or kiln-dried; it is then ground into a coarse powder by two large cylinders of stone, revolving in a vertical position, like those used for crushing linseed-cake; in this state it is washed and subjected to hydrostatic pressure, to free it from the saccharine and other matters which would injure its dyeing qualities. When properly washed, it is again dried, and submitted to the action of another pair of stone cylinders, until it is reduced to an impalpable powder.

The washings of the madder at the dye-stuff factory of Messrs Arthur and Hinshaw were formerly permitted, as in other establishments, to flow into the nearest canal or other reservoir of refuse; but now it is carefully preserved, and distilled into a strong spirit, which, although more volatile and less agreeable to the taste than that distilled from malt or raw grain, may perhaps be found equally useful for various manufacturing purposes.

The process of the fabrication is exceedingly simple: mere washing of the madder supersedes the mashing of the grain or malt in common distilleries. The root being dried and ground, as already stated, is mixed in a series of vats with the requisite proportion of cold water, or water at the ordinary temperature. These vats are 3 feet deep by 5 or 6 in diameter. The mixture is effectually rummaged by the workmen with instruments resembling large hoes, the madder being kept in a state of diffusion in the liquid until it is conceived that the saccharine matter is entirely extracted. The liquor is then drawn off by sluices, and the vats being lined with coarse cloth, it percolates through that medium as a filter, leaving the madder behind, to be again carefully collected, dried, and finally ground for the use of the dyer.

When the madder liquor, or worts, is drawn off, it is let into a kind of under-back, from which it is immediately pumped up into a large fermenting tun: 2 tons of madder are found in practice to yield 2500 gallons of liquor, or madder worts, of a density equivalent to 30° by Allan's saccharometer.

The fermenting tun being filled by the produce of several washings, has usually begun to ferment before it receives the liquor from the last washings. It is not a little remarkable, that this fermentation of the madder liquor commences and proceeds spontaneously, without the addition of yeast or the application of heat. No ferment of any description is added, and the water used throughout the entire process, up to the point of distillation, is at the ordinary temperature. The addition of yeast has been tried, but without any sensible advantage, either in promoting the fermentation or increasing the ultimate yield of spirits. In consequence, however, of the spontaneous character of the process, the fermentation is slower than usual, averaging from six to eight days to a proper attenuation. This is conceived to be accomplished when the gravity of the worts is reduced from 24° to 12°.

From the fermenting tun the wash is immediately run into the still, and the rest of the operation proceeds as in the distillation of malt or raw grain whisky. The still used is STEIN's patent, and is somewhat similar to COFFEY's, but rather more complex in its arrangements, and possessing the undoubted advantage that it may be applied, with equal efficiency, either to the distillation of malt or raw grain whisky. From this still the madder spirit is drawn off at one operation; it may be run weaker or stronger, but is generally taken from the still about 60° to 64° over proof by Sikes' hydrometer.

The produce from 2 tons of madder, yielding, as has been stated, 2500 gallons of liquor at 30°, is about 60 gallons of spirit.

The berries of the *Sorbus Aucuparia* have been used in the North of France for the production of spirit, and the result is said to be equal to the purest distillation from grapes for brandy. The berries, when perfectly ripe, are first exposed to the action of cold in the open air, then put into a wooden vessel, bruised, and boiling water poured on, the menstruum being stirred until it has sunk in temperature to 82° Fahr. (27°·7 C.). A proper quantity of yeast is then added, the whole covered and left to ferment. When the action has terminated, the liquor is put into the still, and drawn over in the usual way. The first running is weak and disagreeable in flavour, but being distilled from very fresh finely-powdered charcoal, in the proportion of 8 or 9 lbs. to 40 gallons of weak spirit, a fine product is obtained. The charcoal should remain in the liquid two or three days before the second distillation.

ALCOHOLOMETRY.—This is the process of ascertaining the centesimal quantity of anhydrous alcohol in a spirituous liquid. It is generally accomplished by determining the specific gravity of the liquid, but it is in this case absolutely necessary that only alcohol and water should be present. The quantity of alcohol in spirit containing much volatile oil or saccharine matter, &c., cannot be at once found by its specific gravity.

It happens sometimes that only small quantities of the alcoholic fluid is at command. In such a case it is impossible to obtain a correct result by determining the specific gravity of the few drops of alcohol obtained by distillation. It is then safer to subject the alcohol to organic analysis by combustion with oxide of copper, and to calculate the quantity of absolute alcohol from the resulting carbonic acid and water. This is of course only applicable when the alcohol does not contain other volatile hydrocarbons.

With the view of being able to calculate the absolute alcohol included in a spirituous liquid from its specific gravity, it was deemed advisable to mix anhydrous alcohol and water in the different proportions, and by experiments, to establish with certainty the specific gravity of these mixtures. Such experiments have been gone through at distinct periods; the most accurate and complete were those performed by GILPIN. They were undertaken by Sir CHARLES BLAGDEN at the instance of the British government, it being of course a matter of great importance to the excise that extreme accuracy should be attained in all determinations, and that their decisions should be above dispute. These determinations were made by GILPIN under Sir CHARLES BLAGDEN's direction, and were first given to the world in 1790. In order to insure their correctness they were subsequently twice repeated, and were ultimately published in the Philosophical Transactions of the Royal Society in 1794.

The method adopted was to weigh the mixture of alcohol and water in a long-necked flask, which was filled up to a mark showing the bulk of a known weight of water. The specific gravity of forty mixtures was thus determined, each one at fifteen different degrees of temperature. Alcohol of specific gravity 0·82514 was used, which was taken as being 0·825, the requisite allowance being made in the tables. From GILPIN's conclusions, aided by results of his own, TRALLES constructed the tables appended.

The percentage of absolute alcohol may be stated by one of two methods; namely, by weight or volume.

Liquors being vended by measure and not by weight, the centesimal amount by volume is usually preferred. But as the bulk of liquids generally, and particularly that of alcohol, increases by heat, it is necessary that their reputed richness should have reference to some normal temperature; this standard, as fixed by Tralles in the construction of his tables, is 60° Fahr.

The percentage by weight remains the same at all temperatures, while the per cent. by volume varies with the temperature of the liquid; and this entails the necessity of having the sample, in the course of being tested, reduced to the standard degree of the table by calculation or otherwise. In the subjoined table water at 39°·8 Fahr., being its maximum density, is taken as the standard for specific gravity, and is put as 1·000, which, at 60° Fahr., equals ·9991.

Tralles' Table I.

Per cent. of alcohol, by volume.	Spec. grav. of the liquid at 60°.	Difference of the spec. gravs.	Per cent. of alcohol, by volume	Spec. grav. of the liquid at 60°.	Difference of the spec. gravs.
0	0·9991		51	0·9315	20
1	9976	15	52	9295	20
2	9961	15	53	9275	20
3	9947	14	54	9254	21
4	9933	14	55	9234	20
5	9919	14	56	9213	21
6	9906	13	57	9192	21
7	9893	13	58	9170	22
8	9881	12	59	9148	22
9	9869	12	60	9126	22
10	9857	12	61	9104	22
11	9845	12	62	9082	22
12	9834	11	63	9059	23
13	9823	11	64	9036	23
14	9812	11	65	9013	23
15	9802	10	66	8989	24
16	9791	11	67	8965	24
17	9781	10	68	8941	24
18	9771	10	69	8917	24
19	9761	10	70	8892	25
20	9751	10	71	8807	25
21	9741	10	72	8842	25
22	9731	10	73	8817	25
23	9720	11	74	8791	26
24	9710	10	75	8765	26
25	9700	10	76	8739	26
26	9689	11	77	8712	27
27	9679	10	78	8685	27
28	9668	11	79	8658	27
29	9657	11	80	8631	27
30	9646	11	81	8603	28
31	9634	12	82	8575	28
32	9622	12	83	8547	28
33	9609	13	84	8518	29
34	9596	13	85	8488	30
35	9583	13	86	8458	30
36	9570	13	87	8428	30
37	9556	14	88	8397	31
38	9541	15	89	8365	32
39	9526	15	90	8332	33
40	9510	16	91	8299	33
41	9494	16	92	8265	34
42	9478	16	93	8230	35
43	9461	17	94	8194	36
44	9444	17	95	8157	37
45	9427	17	96	8118	39
46	9309	18	97	8077	41
47	9391	18	98	8034	43
48	9373	18	99	7988	46
49	9354	19	100	7939	49
50	9335	19			

The third column of this table exhibits the differences of the specific gravities, in order to facilitate the calculation of fractions of percentage for specific gravities intermediate between those stated in the table—the difference in the densities, as given in the third column, becoming the denominator of the fraction, and the variation between the next and greatest specific gravity in the table and that of the liquid in question forming the numerator. To illustrate this method of calculation by an example, let it be supposed that the specific gravity of a liquid was found to be ·9260 at 60° Fahr., which numbers, according to the table, would indicate a percentage of alcohol between 53 and 54, or 53 and a fraction whose numerator is the difference between ·9275, the specific gravity of an alcohol 53 per cent., and ·9260, which difference equals 15; this number forms the numerator of the fraction, whose denominator is the difference between the specific gravities of the liquids containing 53 and 54 per cent. of alcohol, and which in the foregoing table is 21; hence the value of the liquid, specific gravity ·9260, is $53\frac{15}{21}$, or 53·71 per cent.

The content, by weight, of alcohol in a liquid, the centesimal value of which per volume has been found, is ascertained by a simple calculation. This operation is done by multiplying the content per volume of alcohol into the specific gravity of absolute alcohol, and dividing the product by the specific gravity of the liquid. An example will aid the reader in comprehending the manner of performing the work:—Suppose an alcohol of 50 per cent. by volume, whose density, according to the preceding table, is ·9335. Absolute alcohol in the table is ·7939, and this multiplied by 50, and the product divided by ·9335, gives the percentage by weight, thus:—

$$\cdot7939 \times 50 = 39\cdot6950 \div \cdot9335 = 42\cdot5227 \text{ per cent.}$$

It necessarily happens that alcoholic liquors are seldom at the very degree of the thermometer at which the preceding table has been drawn up, and as it is difficult to bring the sample to mark 60° Fahr., Tralles, for the purpose of surmounting this obstacle, constructed another table, wherein the volume of alcohol is given corresponding with the temperature of the liquid at the time of the experiment.

Tralles' Table II.

Per cent., by volume, of absolute alcohol.	Spec. grav. of the liquid at 60°.	Increase of spec. grav. at the indicated temperature *below* 60°.					
		+55°	60°	45°	40°	35°	30°
0	0·9991	4	7	9	9	9	7
5	9919	4	7	9	10	10	9
10	9857	5	9	12	14	15	15
15	9802	6	12	17	21	23	25
20	9751	8	16	23	29	35	39
25	9700	10	21	31	39	48	56
30	9646	13	26	39	51	62	73
35	9583	16	31	46	61	75	89
40	9510	18	35	52	70	87	103.
45	9427	19	39	57	76	94	112
50	9355	20	40	60	80	99	118
55	9234	21	42	63	84	104	124
60	9126	22	43	65	86	107	127
65	9013	22	45	67	88	109	130
70	8892	22	45	68	90	112	133
75	8765	23	46	68	91	113	135
80	8631	23	47	70	92	115	137
85	8488	23	47	70	93	116	139
90	8332	24	48	71	94	117	140

TRALLES' TABLE II.—Concluded.

Per cent., by volume, of absolute alcohol.	Spec. grav. of the liquid at 60°.	Decrease of spec. grav. at the indicated temperature *above* 60°.							
		65°	70°	75°	80°	85°	90°	95°	100°
0	0·9991	1	11	17	24	32	40	50	60
5	9919	1	11	18	25	33	42	51	62
10	9857	6	13	20	29	37	47	57	68
15	9802	7	15	25	34	44	55	67	79
20	9751	9	19	30	41	53	66	79	93
25	9700	11	24	36	50	63	78	93	109
30	9646	14	28	43	59	75	91	108	125
35	9583	17	33	50	68	86	104	122	141
40	9510	18	37	56	75	94	114	136	154
45	9427	20	40	60	80	101	122	143	154
50	9335	21	42	63	84	106	128	150	173
55	9234	22	43	65	87	109	132	155	178
60	9126	22	44	67	90	113	136	159	183
65	9013	22	45	68	92	115	138	162	187
70	8892	23	46	69	93	117	141	165	190
75	8765	23	46	70	94	119	143	167	192
80	8631	23	47	71	96	120	144	169	194
85	8488	24	48	72	96	121	145	170	195
90	8332	24	48	72	97	121	146	171	196

A further objection to these tables is, that the specific gravity of the mixtures of alcohol and water at elevated or reduced temperatures is not the real but the apparent density. The cause of this discrepancy is, that the glass or copper vessel in which the liquids are weighed are liable to expand or contract with change of temperature,

TRALLES constructed a third table, in order that the percentage by volume in an alcoholic liquid might be ascertained from the more uniform arrangement of the numbers denoting the specific gravities.

In Table III. the densities are given of the several mixtures from 30° to 85°, as ascertained by a glass instrument; but these numbers, by the aid of the allowance, as seen in Table IV., can be made to correspond with the indications of a brass alcoholometer.

TRALLES' TABLE III.

Per cent. of alcohol, by volume.	Specific gravity of the liquid, ascertained by glass instruments, at the indicated temperatures.											
	30°	35°	40°	45°	50°	55°	60°	65°	70°	75°	80°	85°
0	·9994	·9997	·9997	·9998	·9997	·9994	·9991	·9987	·9981	·9976	·9970	·9962
5	9924	9926	9926	9925	9925	9922	9919	9915	9909	9903	9897	9889
10	9868	9869	9868	9867	9865	9861	9857	9852	9845	9839	9831	9823
15	9823	9822	9820	9817	9813	9807	9802	9796	9788	9779	9771	9761
20	9786	9782	9777	9772	9766	9759	9751	9743	9733	9723	9713	9701
25	9752	9745	9737	9729	9720	9709	9700	9690	9678	9666	9653	9640
30	9715	9705	9694	9683	9671	9658	9646	9633	9619	9605	9590	9574
35	9668	9655	9641	9627	9612	9598	9583	9567	9551	9535	9518	9500
40	9609	9594	9577	9560	9544	9527	9510	9193	9474	9456	9438	9419
45	9535	9518	9500	9482	9464	9445	9427	9408	9388	9369	9359	9329
50	9449	9431	9413	9393	9374	9354	9335	9315	9294	9274	9253	9232
55	9354	9335	9316	9295	9275	9254	9234	9213	9192	9171	9250	9128
60	9249	9230	9210	9189	9168	9147	9126	9105	9083	9061	9039	9016
65	9140	9120	9099	9078	9056	9034	9013	8992	8969	8947	8924	8901
70	9021	9001	8980	8958	8936	8913	8892	8870	8847	8825	8801	8778
75	8896	8875	8854	8832	8810	8787	8765	8743	8720	8697	8673	8619
80	8764	8743	8721	8699	8676	8653	8631	8609	8585	8562	8538	8514
85	8623	8601	8579	8556	8533	8510	8488	8465	8441	8418	8394	8370
90	8469	8446	8423	8401	8379	8355	8332	8309	8285	8262	8238	8214

TRALLES' TABLE IV.

To be subtracted.						To be added.					
30°	35°	40°	45°	50°	55°	60°	65°	70°	75°	80°	85°
·0005	·0004	·0003	·0002	·0002	·0001	—	·0001	·0002	·0002	·0003	·0004

The necessity for using Table IV. arises from the discrepancy in the contraction or expansion of glass or brass at different temperatures, occasioning variation in the density; hence these numbers must be added or subtracted, as directed, according to the temperature, when a brass instrument is employed.

It occurs that liquids are tested whose temperature and specific gravity are intermediate between those instanced in the preceding table, and in this case the corresponding content of alcohol must be found by calculation. To give an example:—Let it be supposed that the specific gravity of an alcoholic solution has been found by experiment to be ·9320 at a temperature of 72° Fahr. In the column under 70°, the specific gravity ·9388 is that which is next over the above number, and in the column under 75° the gravity nearest to it is ·9369, both of which exceed ·9320—the former by ·0068, and the latter by ·0049. The difference between these two numbers is ·0019, which is the variation for 5°, or between 70° and 75°, and by dividing by 5, the quotient is $\frac{\cdot0019}{5}$, the difference for each degree. As the temperature of the liquid under examination is 72°, or 2° over 70°, it is necessary to reduce the specific gravity to what it would indicate were the liquid at 70°, by adding a multiple of $\frac{\cdot0019}{5}$ by 2, or $\frac{\cdot0038}{5} = \cdot0007\frac{3}{5}$ to the specific gravity obtained—·9320—which would make it $\cdot9327\frac{3}{5}$, or omitting fractions, ·9327. By referring to the table, will be seen in the horizontal column opposite 45 per cent. of alcohol, and under 70°, the specific gravity ·9388, and in the same column, in a line with 50 per cent. of spirit, ·9294, whose difference from the foregoing is ·0094; thus the variation in gravity at the same temperature, for 5 per cent. of alcohol, is ·0094. In like manner, the difference of ·9388 and ·9327, the reduced gravity for the liquid specified, is ·0061; and since 5 per cent. of spirit has been shown to correspond with ·0094, it is found by simple proportion that ·0061 will be equivalent to 3·24 per cent., and this, added to 45 per cent., the spirit in the liquid of gravity next preceding that which is supposed to be ascertained, gives 48·24 as the percentage by volume of alcohol. Numerically—

$$·9388 - ·9320 = ·0068, \text{ and}$$
$$·9369 - ·9320 = ·0049.$$

By subtracting the lesser of these from the greater, namely, ·0068 — ·0049 = ·0019, the difference for the 5° between 70° and 75° Fahr., giving for each degree $\frac{·0019}{5}$; and to reduce the corresponding gravity of the liquid at 72° to that of 70°, there must be added to it $\frac{·0019}{5} \times 2\ (= 72 - 70) = \frac{·0038}{5} = ·0007\frac{3}{5}$; and thus one obtains $·9320 + ·0007\frac{3}{5} = ·9327\frac{3}{5}$, or omitting fractions, ·9327. Again,

$$·9388 - ·9294 = ·0094, \text{ and}$$
$$·9388 - ·9327 = ·0061.$$

And since the former indicates a difference of 5 per cent. in the amount of alcohol, at the same temperature, the equivalent for ·0061 under similar circumstances is obtained by simple proportion:—

As 94 : 5 :: 61 : 3·24, which, when added to 45, gives 48·24, the content per cent. by volume of alcohol in the liquid. From all the tables in the foregoing for the determination of the volume of alcohol in a liquid, at whatever temperature it may stand, it is seen that reference is always had to the percentage at the normal temperature of 60° Fahr. Hence, in examining an alcoholic liquid for its true content of pure alcohol, the heat must be diminished to 60°, or else the temperature noted at the time of experiment, and then by calculation reduced to 60°; or the content of alcohol at 60° may be found as in Table II., where the specific gravity at the different temperatures is in inverse proportion to the volume of alcohol.

To avoid this calculation, Tralles calculated another table, which gives the content in volume of absolute alcohol in a liquid, reference being had to the bulk of the liquid at the temperature at which it is measured.

For the prevention of error, it is absolutely necessary to determine the specific gravity of the liquid at the same degree at which it stands when measured; a glass instrument should be employed.

Tralles' Table V.

To ascertain at any temperature, from the specific gravity, the quantity of absolute alcohol in a liquid expressed in volume centesimally, at the indicated temperature.

Per cent. of absolute alcohol in the liquid as measured.	Specific gravity of the liquid, ascertained by glass instruments, at the indicated temperatures.											
	30°	35°	40°	45°	50°	55°	60°	65°	70°	75°	80°	85°
0	0·9994	·9997	·9997	·9998	·9997	·9994	·9991	·9987	·9981	·9976	·9970	·9962
5	9924	9926	9926	9926	9925	9922	9919	9915	9909	9903	9897	9889
10	9868	9869	9868	9867	9865	9861	9857	9852	9845	9839	9831	9823
15	9823	9822	9820	9817	9813	9807	9802	9796	9788	9779	9771	9761
20	9786	9782	9777	9772	9766	9759	9751	9743	9733	9722	9711	9700
25	9753	9746	9738	9729	9720	9709	9700	9690	9678	9665	9652	9638
30	9717	9707	9695	9684	9672	9659	9646	9632	9618	9603	9588	9572
35	9671	9658	9644	9629	9614	9599	9583	9566	9549	9532	9514	9495
40	9615	9598	9581	9563	9546	9528	9510	9491	9472	9452	9433	9412
45	9514	9525	9506	9486	9467	9447	9427	9406	9385	9364	9342	9320
50	9460	9440	9120	9399	9378	9356	9335	9313	9290	9267	9244	9221
55	9368	9347	9325	9302	9279	9256	9231	9211	9187	9163	9139	9114
60	9267	9245	9222	9198	9174	9150	9126	9102	9076	9051	9026	9000
65	9162	9138	9113	9088	9063	9038	9013	8988	8962	8936	8909	8882
70	9046	9021	8996	8970	8944	8917	8892	8866	8839	8812	8784	8756
75	8925	8890	8873	8847	8820	8792	8765	8738	8710	8681	8652	8622
80	8798	8771	8714	8716	8688	8659	8631	8602	8573	8544	8514	8483
85	8663	8635	8606	8577	8547	8517	8488	8458	8427	8396	8365	8333
90	8517	8486	8455	8425	8395	8363	8332	8300	8268	8236	8201	8171

Tralles' Table VI.

To be added.						To be subtracted.					
30°	35°	40°	45°	50°	55°	60°	65°	70°	75°	80°	85°
·0005	·0004	·0003	·0002	·0002	·0001	—	·0001	·0002	·0002	·0003	·0004

For the reasons assigned under Tables III. and IV., the numbers in Table VI. must be added or subtracted, as may be deemed necessary, to or from the specific gravities given in Table V., according to the temperature.

From the above tables, the true amount of alcohol in a liquor, or "its richness," may be ascertained by the specific gravity.

It will be as well to consider here the method used for ascertaining the specific gravity of the various liquids. The density is invariably found by one of two methods—either by actually weighing a portion of the liquor in an accurate specific gravity bottle, or by hydrometer.

The latter mode is generally adopted, being more expeditious in practice. Various hydrometers are employed, the principal ones being Beaumé's, Sikes', and Dicas', all of which have corresponding tables to indicate the quantities of alcohol according to the gravity. It being thought more convenient and simple to have an instrument which would indicate at once the amount of alcohol, one was formed, partly on the plan of the hydrometer, to which the term *alcoholometer* has been applied; it may be made either of glass or brass. TRALLES, in constructing one of these instruments, has drawn up the annexed alcoholometric table for guidance, which shows, from the portion of the stem immersed in the liquor, the amount of alcohol contained at 60° Fahr.

TRALLES' TABLE VII.

Per cent. alcohol, by volume.	Length of immersed part of stem.	Distance between degrees of scale indicating per cent.	Per cent. alcohol, by volume.	Length of immersed part of stem.	Distance between degrees of scale indicating per cent.
0	9		51	735	23
1	24	15	52	758	23
2	39	15	53	782	24
3	54	15	54	806	24
4	68	14	55	830	24
5	82	14	56	854	24
6	95	13	57	879	25
7	108	13	58	905	26
8	121	13	59	931	26
9	133	12	60	957	26
10	145	12	61	984	27
11	157	12	62	1011	27
12	169	12	63	1039	28
13	180	11	64	1067	28
14	191	11	65	1096	29
15	202	11	66	1125	29
16	213	11	67	1154	29
17	224	11	68	1184	30
18	235	11	69	1215	31
19	245	10	70	1246	31
20	256	10	71	1278	32
21	266	10	72	1310	32
22	277	11	73	1342	32
23	288	11	74	1375	33
24	299	11	75	1409	34
25	310	11	76	1443	34
26	321	11	77	1478	35
27	332	11	78	1514	36
28	344	12	79	1550	36
29	355	11	80	1587	37
30	367	12	81	1624	37
31	380	13	82	1662	38
32	393	13	83	1701	39
33	407	14	84	1740	39
34	420	13	85	1781	41
35	434	14	86	1823	42
36	449	15	87	1866	43
37	465	16	88	1910	44
38	481	16	89	1955	45
39	498	17	90	2002	47
40	515	17	91	2050	48
41	533	18	92	2099	49
42	551	18	93	2150	51
43	569	18	94	2203	53
44	588	19	95	2259	56
45	608	20	96	2318	59
46	628	20	97	2380	62
47	648	20	98	2447	67
48	669	21	99	2519	72
49	690	21	100	2597	78
50	712	22			

In the practical application of the preceding table, when graduating the alcoholometer, it is requisite to have two liquids of a standard strength and temperature.

Distilled water may be one of these, and the other any alcoholic liquor, the percentage of which has been precisely determined, but it is necessary that both should be at 60° Fahr. The level at which the instrument stands in each should be scratched on the stem, and the intermediate space accurately divided according to the strength of the liquid. If the alcoholic liquor be 49 per cent., and the difference between the point to which the instrument sinks in this liquid and water be divided into 690 — 9 = 681 equal parts, the addition of 22 such parts will indicate the point for 50 per cent., and 23 more for 51 per cent., and so on, as is seen in the third column in the table. By adding 9 such divisions below zero, or the water-level mark, the point is attained from which the numbers in the second column in the table are calculated for each succeeding per cent. of spirit.

In graduating the scale, it would perhaps be more convenient to have two instruments; one to have indications denoting a percentage, say from 1 to 80, and the other from the latter up to pure alcohol. For this end it would, however, be absolutely necessary to adjust the instrument having the higher indications, with two liquids at the normal degree of temperature of the preceding, whose percentage of alcohol should be 80 and 100; then, to observe the points to which it sinks in these, and divide the intermediate space into measures corresponding with those in the table.

In constructing this alcoholometer it is essential that its stem should for ordinary purposes be as uniform as possible. Tubes which do not vary throughout their length more than one-thirtieth of their diameter, may be safely used.

The temperature of the liquid must be at 60° Fahr., or else the true percentage must be calculated from this gravity in the usual way, by the preceding Tables III. or IV.

To dispense with the trouble of such a calculation, TRALLES devised another table, by which the real percentage of alcohol is found in liquids of different temperatures, from the results exhibited by the instrument. This table is given below, Table VIII., and corresponds with the preceding Table III.

The numbers in the vertical columns under the temperatures, are the observed degrees of the alcoholometer, and indicate the percentage of the absolute alcohol by volume. If an alcoholic liquid at a temperature of 75° Fahr. be found to contain 62·9 per cent. by volume, by reference to the table its true percentage at 60° Fahr. is 60.

Tables IX. and X. following give the richness per cent. by volume of the liquid at the temperature it possesses when tested. They correspond with Table V., and like it require that the solution should be tested at exactly the same temperature at which it is measured. Table IX. is for a glass, and Table X. for a brass alcoholometer.

TRALLES' TABLE VIII.

To find the true percentage of absolute alcohol by volume, in a liquid at 60° Fahr. from the observed percentago indicated by a glass alcoholometer at any other temperature (degrees Fahr.).

30°	35°	40°	45°	50°	55°	60°	60°	65°	70°	75°	80°	85°
— 0·2	— 0·4	— 0·4	— 0·5	— 0·4	— 0·2	0	0	+ 0·2	+ 0·6	+ 1·0	+ 1·4	+ 1·9
+ 4·6	+ 4·5	+ 4·5	+ 4·5	+ 4·6	+ 4·8	5	5	5·3	5·8	6·2	6·7	7·3
9·1	9·0	9·4	9·2	9·3	9·7	10	10	10·4	11·0	11·6	12·3	13·0
13·0	13·1	13·3	13·5	13·9	14·5	15	15	15·6	16·3	17·1	18·0	19·0
16·5	16·9	17·4	17·8	18·5	19·2	20	20	20·8	21·8	22·8	23·8	24·9
19·9	20·6	21·4	22.2	23·0	24·1	25	25	25·9	27·0	28·2	29·4	30·5
23·5	24·5	25·7	26·6	27·7	28·8	30	30	31·1	32·2	33·4	34·5	35·7
28·0	29·2	30·4	31·6	32·7	33·8	35	85	36·2	37·3	38·4	39·5	40·6
33·0	34·2	35·4	36·7	37·8	39·0	40	40	41·1	42·2	43·3	44·3	45·4
38·4	39·6	40·7	41·8	42·9	43·9	45	45	46·1	47·1	48·2	49·2	50·3
43·7	44·7	45·8	46·9	47·9	49·0	50	50	51·0	52·0	53·0	54·0	55·1
49·0	50·0	51·0	52·0	53·0	54·0	55	55	54 9	56·9	57·9	58·9	59·9
54·2	55·2	56·2	57·1	58·1	59·0	60	60	60·9	61·9	62·9	63·8	64·9
59·4	60·3	61·2	62·2	63·1	64·0	65	65	65·9	66·8	67·7	68·6	69·6
64·6	65·5	66·4	67·3	68·2	69·1	70	70	70·8	71·7	72·6	73·5	74·5
69·8	70·7	71·5	72·4	73·3	74·2	75	75	75·8	76·7	77·6	78·4	79·3
75·0	75·8	76·6	77·5	78·4	79·2	80	80	80·8	81·7	82·4	83·2	84·1
80·3	81·1	81·8	82·6	83·5	84·3	85	85	85·7	86·5	87·3	88·0	88·8
85·6	86·4	87·1	87·9	88·6	89·3	90	90	90·7	91·4	92·0	92·7	93·4

TRALLES' TABLE IX.

To find the true percentage of absolute alcohol by volume, in a liquid of any temperature, from the observed per centage indicated by the glass alcoholometer at the same temperature.

True per cent. of alcohol by volume, at 60° Fahr.	Observed per cent. indicated by the glass alcoholometer.									
	30°	35°	40°	45°	50°	55°	65°	70°	75°	80°	85°
0	— 0·2	— 0·4	— 0·4	— 0·5	— 0·4	— 0·2	+ 0·2	+ 0·6	+ 1·0	+ 1·4	+ 1·9
5	+ 4·6	+ 4·5	+ 4·5	+ 4·5	+ 4·6	+ 4·8	5·3	5·8	6·2	6·7	7·3
10	9·1	9·0	9·1	9·2	9·3	9·7	10·4	11·0	11·6	12·3	13·0
15	13·0	13·1	13·3	13·6	14·1	14·5	15·6	16·3	17·1	18·0	19·0
20	16·5	16·9	17·4	17·9	18·5	19·2	20·8	21·8	22·9	23·9	25·0
25	19·8	20·5	21·3	22·2	23·0	24·1	25·9	27·1	28·3	29·5	30 7
30	23·3	24·3	25·5	26·5	27·6	28·8	81·2	32·3	33·5	84·6	35·9
35	27·7	28·9	30·2	31·4	32·6	33·8	36·3	37·5	38·6	39·7	40·9
40	32·5	33·8	35·1	36·5	37·7	38·9	41·2	42·4	43·5	44·6	45·8
45	37·8	39·1	40·3	41·5	42·7	43·8	46·2	47·3	48·5	49·6	50·8
50	43·1	44·2	45·4	46·6	47·7	48·9	51·1	52·2	53·4	54·5	55·6
55	48·3	49·4	50·5	51·6	52·8	53·9	56·1	57·2	58·3	59·4	60·5
60	53·4	54·5	55·6	56·7	57·8	58·9	61·1	62·2	63·3	64·4	65·5
65	58·4	59·5	60·6	61·7	62·8	63·9	66·0	67·1	68·2	69·3	70·4
70	63·5	64·6	65·7	66·8	67·9	69·0	71·0	72·1	73·2	74·3	75·4
75	68·6	69·7	70·7	71·8	72·9	74·0	76·0	77·1	78·2	79·2	80·3
80	73·7	74·8	75·8	76·9	78·0	79·0	81·0	82·1	83·1	84·1	85·2
85	78·8	79·8	80·9	81·9	83·0	84·0	86·0	87·0	88·0	89·0	90·0
90	84·0	85·1	86·1	87·1	88·1	89.1	91·0	91·9	92·8	93·7	94·6

Thus, if the alcoholometer indicated 59·4 per cent. in a liquid at 80°, the table would give its true percentage as 55 per cent., that is, 100 volumes of the liquid at 80° contain 55 volumes of anhydrous alcohol.

TRALLES' TABLE X.

To find the true percentage of absolute alcohol in a liquid of any temperature, from the observed percentago indicated by a brass alcoholometer at the same temperature.

True per cent. of alcohol by volume.	Observed per cent. indicated by the brass alcoholometer.									
	30°	35°	40°	45°	50°	55°	65°	70°	75°	80°	85°
0	— 0·1	— 0·1	— 0·2	— 0·3	— 0·3	— 0·2	+ 0·2	+ 0·5	+ 0·9	+ 1·2	+ 1·7
5	+ 5·0	+ 4·8	+ 4·7	+ 4·8	+ 4·7	+ 4·8	5·2	5·6	6·1	6·5	7·0
10	9·5	9·4	9·4	9·4	9·5	9·7	10·3	10·8	11·4	12·0	12·6
15	13·5	13·5	13·6	13·7	14·0	14·6	15·5	16·2	17·0	17·7	18·6
20	17·0	17·3	17·7	18·1	18·7	19·3	20·7	21·6	22·7	23·7	24·0
25	20·3	20·9	21·6	22·4	23·3	24·2	25·8	26·9	28·1	29·2	30·3
30	23·8	24·7	25·8	26·8	27·8	28·9	31·1	32·2	33·3	34·4	35·5
35	28·2	29·3	30·4	31·6	32·8	33·9	36·2	37·3	38·4	39·5	40·7
40	32·9	34·1	35·4	36·7	37·9	39·0	41·1	42·2	43·4	44·5	45·6
45	38·1	39·3	40·4	41·6	42·7	43·9	46·1	47·2	48·3	49·4	50·5
50	43·4	44·5	45·6	46·7	47·8	48·9	51·1	52·2	53·3	54·4	55·5
55	48·5	49·6	50·7	51·8	52·9	54·0	56·0	57·1	58·2	59·3	60·4
60	53·6	54·6	55·7	56·8	57·8	58·9	61·0	62·1	63·2	64·3	65·3
65	58·6	59·7	60·7	61·8	62·8	63·9	66·0	67·1	68·1	69·2	70·2
70	63·7	64·8	65·8	66·9	67·9	69·0	71·0	72·1	73·1	74·2	75·2
75	68·8	69·8	70·9	74·9	72·9	74·0	76·0	77·0	78·1	79·1	80 1
80	73·9	74·9	75·9	76·9	78·0	79·0	81·0	82·0	83·0	84·0	85·0
85	79·0	80·0	81·0	82·0	83·0	84·0	86·0	87·0	88·0	88·9	89·9
90	84·2	85·3	86·2	87·2	88·1	89.1	90·9	91·9	92·8	93·7	94·5

The preceding ten tables of Tralles contain all that is requisite for the determination of the volume of alcohol in a liquid; they have been constructed on the principle of the tables of Gilpin. Tralles experiments were made in 1811, he found that Gilpin's alcohol, so far from being absolute, contained 10·8 per cent. of water. Since then further extensive researches have been made by many eminent chemists, the most important being those of Gay-Lussac, who in 1824 drew up much more extensive tables, which require scarcely any interpolation.

Gay-Lussac's instrument is like a glass hydrometer, the stem of which is divided into degrees like that of Tralles, to indicate the percentage of alcohol by volume, but the temperature at which the graduation was made was 15° C. or 59° Fahr., instead of 60° Fahr., as the normal temperature of Tralles' tables. Water is taken as unity at this temperature. The stem of the instrument is divided, from the point at which it stands in water, into 100 divisions, so that each on the scale is equal to one per cent. of alcohol; the 100th division indicates pure or absolute alcohol, while 0 or zero equals pure water at 15 C.

The instrument, if immersed in an alcoholic liquor at 59° Fahr., marks the strength by the number of degrees below the surface; thus, if the alcoholometer stands at 57 in a liquid at 59°, such a solution contains 57 per cent. by volume of alcohol.

In consequence of the temperature 59° Fahr. (15° C.) being taken by Gay-Lussac, as the fundamental numbers for determining the relation, the percentage of alcohol and the specific gravities are in a slight degree different from those of Tralles, but the variation is so minute that in practice it may be overlooked.

In the annexed table the fundamental numbers of the centesimal amount per volume, together with the specific gravities of the different alcoholic mixtures at 59° Fahr. (15° C.), are given:—

Per cent. of alcohol by volume.	Specific gravity of the liquid at 59°.	Per cent. of alcohol by volume.	Specific gravity of the liquid at 59°.
100	0·7947	60	0·9141
95	0·8168	55	0·9248
90	0·8346	50	0·9348
85	0·8502	45	0·9440
80	0·8645	40	0·9523
75	0·8799	35	0·9595
70	0·8907	10	0·9656
65	0·9027	0	1·0000

The first table by Gay-Lussac is denominated *Table for the* REAL *strength of spirits*, and corresponds with Table VIII. of Tralles. This table gives the true percentage by volume at 59° Fahr. (15° C.) from the observed percentage of the alcoholometer at any other temperature. Two numbers are placed in the same horizontal column, and vertical to each other: those in the top are the observed centesimal quantities of the alcoholometer, while the large figures in the vertical column below them give the real percentages at 59° Fahr. when tested at the temperature found in the left-hand vertical column. The numbers in small figures beneath the real percentages denote the bulk at 59° Fahr. (15° C.) of 1000 measures of liquid; this number, multiplied by the real percentage under which it stands, gives the absolute quantity of alcohol at the temperature at which the experiment is performed.

To read the table, suppose an alcoholic liquid of 50 strength indicated by the alcoholometer at a temperature of 68° Fahr., the observed percentage—50—is sought in the horizontal column at the top; in the vertical column beneath this and on a line with 68°, in the temperature column at the left hand, will be found the number 48·2, which is the quantity of *real* spirit at 59°. The number 996, in small figures below the indicated strength at 59°, is the bulk or volume which 1000 volumes would occupy at the standard temperature; and by multiplying this number by the proper strength, and dividing the product by 1000, the strength or true volume of spirit in 1000 of the liquid is ascertained; thus—

996 + 48·2 = 48007·2 + 1000 = 48·00, the percentage;

hence, 100 volumes of the liquor at 58° contain 48 volumes of absolute alcohol at 59°.

Morazeau constructed, with one of Gay-Lussac's alcoholometers, a table somewhat more in detail, which differs slightly from the preceding in the figures denoting the density, although they have reference to the same temperature.

When, however, the liquors to be tested stand at a higher degree than 59° Fahr. (15° C.), no provision is made to discover the true amount of spirit, which must then be sought either by other tables or by the calculation given earlier.

Per cent. of alcohol by volume.	Specific gravity.	Per cent. of alcohol by volume.	Specific gravity.	Per cent. of alcohol by volume.	Specific gravity.
0	1·000	34	0·962	68	0·896
1	0·999	35	0·960	69	0·893
2	0·997	36	0·959	70	0·891
3	0·996	37	0·957	71	0·888
4	0·994	38	0·956	72	0·884
5	0·993	39	0·954	73	0·881
6	0·992	40	0·953	74	0·879
7	0·990	41	0·951	75	0·876
8	0·989	42	0·949	76	0·874
9	0·988	43	0·948	77	0·871
10	0·987	44	0·946	78	0·868
11	0·986	45	0·945	79	0·865
12	0·984	46	0·943	80	0·863
13	0·983	47	0·941	81	0·860
14	0·982	48	0·940	82	0·857
15	0·981	49	0·938	83	0·854
16	0·980	50	0·936	84	0·851
17	0·979	51	0·934	85	0·848
18	0·978	52	0·932	86	0·845
19	0·977	53	0·930	87	0·842
20	0·976	54	0·928	88	0·838
21	0·975	55	0·926	89	0·835
22	0·974	56	0·924	90	0·832
23	0·973	57	0·922	91	0·829
24	0·972	58	0·920	92	0·826
25	0·971	59	0·918	93	0·822
26	0·970	60	0·915	94	0·818
27	0·969	61	0·913	95	0·814
28	0·968	62	0·911	96	0·810
29	0·967	63	0·909	97	0·805
30	0·966	64	0·906	98	0·800
31	0·965	65	0·904	99	0·795
32	0·964	66	0·902	100	0·747
33	0·963	67	0·899		

(GAY-LUSSAC).—ALCOHOLOMETRIC TABLE I.

To find the per cent., by volume, in a liquid at 59° from the observed per cent. at any other temperature.
The temperature Centigrade is below that of Fahrenheit.

Temp. Fahr.	Observed per centage of the Alcoholometer.										Temp Fahr.	Observed per centage of the Alcoholometer.									
	1 p.cent.	2 p cent.	3 p.cent.	4 p.cent.	5 p.cent.	6 p.cent.	7 p.cent.	8 p.cent.	9 p.cent.	10 p.cent		11 p.cent.	12 p.cent.	13 p.cent.	14 p.cent.	15 p.cent.	16 p.cent.	17 p.cent.	18 p.cent.	19 p.cent.	20 p.cent.
32·0° 0° C.	1·3 1000	2·4 1000	3·4 1000	4·4 1000	5·4 1000	6·5 1001	7·5 1001	8·6 1001	9·7 1001	10·9 1001	32·0° 0° C.	12·2 1001	13·4 1002	14·7 1002	16·1 1002	17·5 1002	18·9 1003	20·3 1003	21·6 1004	22·9 1004	24·2 1004
33·8 1 C.											33·8 1 C.		13·4 1002	14·7 1002	16 1002	17·3 1002	18·7 1003	20 1003	21·3 1003	22·6 1004	23·9 1004
35 6 2 C.											35·6 2 C.		13·4 1002	14·7 1002	16 1002	17·2 1002	18·5 1003	19·8 1003	21·1 1003	22·3 1004	23·6 1004
37·4 3 C.											37·4 3 C.		13·3 1001	14·6 1002	15·9 1002	17·1 1002	18·3 1002	19·6 1003	20·8 1003	22 1003	23·3 1004
39·2 4 C.											39·2 4 C.		13·3 1001	14·5 1002	15·8 1002	16·9 1002	18·1 1002	19·4 1002	20·6 1003	21·8 1003	23 1003
41·0 5 C.	1·4 1001	2·5 1001	3·5 1001	4·5 1001	5·5 1001	6·6 1001	7·7 1001	8·7 1001	9·8 1001	10·9 1001	41·0 5 C.	12·1 1001	13·2 1001	14·4 1001	15·7 1002	16·8 1002	18 1002	19·2 1002	20·4 1003	21·5 1003	22·7 1003
42·8 6 C.											42·8 6 C.		13·1 1001	14·3 1001	15·6 1002	16·7 1002	17·8 1002	19 1002	20·2 1003	21·3 1003	22·4 1003
44·6 7 C.											44·6 7 C.		13 1001	14·2 1001	15·4 1001	16·6 1002	17·7 1002	18·8 1002	20 1002	21 1002	22·1 1002
46·4 8 C.											46·4 8 C.		13 1001	14·1 1001	15·3 1001	16·4 1001	17·5 1001	18·6 1002	19·7 1002	20·7 1002	21·8 1002
48·2 9 C.											48·2 9 C.		12·9 1001	14 1001	15·1 1001	16·2 1001	17·3 1001	18·4 1001	19·5 1001	20 5 1002	21·6 1002
50·0 10 C.	1·4 1000	2·4 1000	3·4 1001	4·5 1001	5·5 1001	6·5 1001	7·5 1001	8·5 1001	9·5 1001	10·6 1001	50·0 10 C.	11·7 1001	12·7 1001	13·8 1001	14·9 1001	16 1001	17 1001	18·1 1001	19·2 1001	20·2 1001	21·3 1001
51·8 11 C.	1·3 1000	2·4 1000	3·4 1000	4·4 1001	5·4 1001	6·4 1001	7·4 1001	8·4 1001	9·4 1001	10·5 1001	51·8 11 C.	11·6 1001	12·6 1001	13·6 1001	14·7 1001	15·8 1001	16·8 1001	17·9 1001	19 1001	20 1001	21 1001
53·6 12 C.	1·2 1000	2·3 1000	3·3 1000	4·3 1000	5·3 1000	6·3 1000	7·3 1000	8·3 1000	9·3 1000	10·4 1000	53·6 12 C.	11·5 1000	12·5 1001	13·5 1001	14·6 1001	15·6 1001	16·6 1001	17·6 1001	18·7 1001	19·7 1001	20·7 1001
55·4 13 C.	1·2 1000	2·2 1000	3·2 1000	4·2 1000	5·2 1000	6·2 1000	7·2 1000	8·2 1000	9·2 1000	10·3 1000	55·4 13 C.	11·4 1000	12·4 1000	13·4 1000	14·4 1000	15·4 1000	16·4 1000	17·4 1000	18·5 1000	19·5 1000	20·5 1000
57·2 14 C.	1·1 1000	2·1 1000	3·1 1000	4.1 1000	5·1 1000	6·1 1000	7·1 1000	8·1 1000	9·1 1000	10·2 1000	57·2 14 C.	11·2 1000	12·2 1000	13·2 1000	14·2 1000	15·2 1000	16·2 1000	17·2 1000	18·2 1000	19·2 1000	20·2 1000
59·0 15 C.	1 1000	2 1000	3 1000	4 1000	5 1000	6 1000	7 1000	8 1000	9 1000	10 1000	59·0 15 C.	11 1000	12 1000	13 1000	14 1000	15 1000	16 1000	17 1000	18 1000	19 1000	20 1000
60·8 16 C.	0·9 1000	1·9 1000	2·9 1000	3·9 1000	4·9 1000	5·9 1000	6·9 1000	7·9 1000	8·9 1000	9·9 1000	60·8 16 C.	10·9 1000	11·9 1000	12·9 1000	13·9 1000	14·9 1000	15·9 1000	16·9 1000	17·8 1000	18·7 1000	19·7 1000
62·6 17 C.	0·8 1000	1·8 1000	2·8 1000	3·8 1000	4·8 1000	5·8 1000	6·8 1000	7·8 1000	8·8 1000	9·8 1000	62·6 17 C.	10·8 1000	11·7 1000	12·7 1000	13·7 1000	14·7 1000	15·6 1000	16·6 1000	17·5 1000	18·4 999	19·4 999
64·4 18 C.	0·7 1000	1·7 1000	2·7 1000	3·7 1000	4·7 1000	5·7 1000	6·7 1000	7·7 1000	8·7 1000	9·7 1000	64·4 18 C.	10·7 1000	11·6 1000	12·5 999	13·5 999	14·5 999	15·4 999	16·3 999	17·3 999	18·2 999	19·1 999
66·2 19 C.	0·6 999	1·6 999	2·6 999	3·5 999	4·5 999	5·5 999	6·5 999	7·5 999	8·5 999	9·5 999	66·2 19 C.	10·5 999	11·4 999	12·4 999	13·3 999	14·3 999	15·2 999	16·1 999	17 999	17·9 999	18·8 999
68·0 20 C.	0·5 999	1·5 999	2·4 999	3·4 999	4·4 999	5·4 999	6·4 699	7·3 999	8·3 999	9·3 999	68·0 20 C.	10·3 999	11·2 999	12·2 999	13·1 999	14 999	14·9 999	15·8 999	16·7 999	17·6 999	18·5 999
69·8 21 C.	0·4 999	1·4 999	2·3 999	3·3 999	4·3 999	5·2 999	6·2 999	7·1 999	8·1 999	9·1 999	69·8 21 C.	10·1 999	11 999	11·9 999	12·8 999	13·7 999	14·6 999	15·5 998	16·4 998	17·3 998	18·2 998
71·6 22 C.	0·3 999	1·3 999	2·2 999	3·2 999	4·1 999	5·1 999	6·1 999	7 999	7·9 999	8·9 999	71·6 22 C.	9·9 999	10·8 999	11·7 999	12·6 998	13·5 998	14·4 998	15·3 998	16·2 998	17 998	17·9 998
73·4 23 C.	0·1 999	1·1 999	2·1 999	3·1 999	4 999	4·9 599	5·9 999	6·8 998	7·8 998	8·7 998	73·4 23 C.	9·7 998	10·6 998	11·5 998	12·4 998	13·3 998	14·1 998	15 998	15·9 998	16·7 998	17·6 998
75·2 24 C.		1 998	1·9 998	2·9 998	3·8 998	4·8 998	5·8 998	6·7 998	7·6 998	8·5 998	75·2 24 C.	9·5 998	10·4 998	11·3 998	12·2 998	13·1 998	13·9 998	14·8 998	15·7 998	16·5 997	17·4 997
77·0 25 C.		0·8 998	1·7 998	2·7 998	3·6 998	4·6 998	5·5 998	6·5 998	7·4 998	8·3 998	77·0 25 C.	9·3 998	10·2 998	11·1 998	12 998	12·8 998	13·6 998	14·5 997	15·4 997	16·2 997	17·1 997
78·8 26 C.		0·7 998	1·6 998	2·6 998	3·5 998	4·4 998	5·4 998	6·3 998	7·2 998	8·1 998	78·8 26 C.	9 998	9·9 997	10·8 997	11·7 997	12·6 997	13·4 997	14·2 997	15·1 997	15·9 997	16·8 997
80·6 27 C.		0·5 998	1·5 998	2·4 998	3·3 998	4·3 998	5·2 998	6·1 998	7 998	7·9 998	80·6 27 C.	8·8 997	9·7 997	10·6 997	11·5 997	12·3 997	13·1 997	14 997	14·8 997	15·6 997	16·5 997
82·4 28 C.		0·3 997	1·3 997	2·2 997	3·1 997	4·1 997	5 997	5·9 497	6·8 997	7·7 997	82·4 28 C.	8·6 997	9·5 997	10·3 997	11·2 997	12 967	12·8 996	13·7 996	14·5 996	15·3 996	16·1 996
81·2 29 C.		0·1 997	1·1 997	2 997	2·9 997	3·9 997	4·8 997	5·7 997	6·6 997	7·5 697	84·2 29 C.	8·4 997	9·2 997	10·1 997	11 997	11·8 996	12·6 996	13·4 996	14·2 996	15 996	15·8 996
86·0 30 C.		0·0 997	0·9 997	1·9 997	2·8 997	3·7 997	4·6 997	5·5 997	6·4 997	7·3 997	86·0 30 C.	8·1 997	9 996	9·8 996	10·7 996	11·5 996	12·3 996	13·1 996	13·9 996	14·7 996	15·5 996

(Gay-Lussac).—Table I.—Continued.

Temp. Fahr.	Observed per centage of the Alcoholometer.									
	21 p.cent.	22 p.cent.	23 p.cent.	24 p.cent.	25 p cent.	26 p.cent.	27 p cent.	28 p.cent.	29 p cent.	30 p.cent.
32·0° 0° C.	25.6 1005	27 1005	28·4 1006	29·7 1006	30·9 1007	32·1 1007	33·2 1007	34·3 1008	35·3 1008	36·3 1008
33·8 1 C.	25·3 1005	26·7 1005	28 1005	29·2 1006	30·4 1006	31·6 1006	32·7 1007	33·8 1007	34·8 1007	35·8 1008
35·6 2 C.	24·9 1004	26·3 1005	27·5 1005	28·8 1005	30 1006	31·2 1006	32·3 1006	33·3 1006	34·4 1007	35·4 1007
37·4 3 C.	24·6 1004	25·9 1005	27·1 1005	28·4 1005	29·6 1005	30·8 1006	31·9 1006	32·9 1006	33·9 1007	34·9 1007
39·2 4 C.	24·3 1001	25·6 1001	26·8 1005	28 1005	29·2 1005	30·4 1005	31·4 1005	32·5 1005	33 5 1006	34·5 1006
41·0 5 C.	24 1003	25·2 1003	26·4 1004	27·6 1004	28·8 1004	30 1001	31 1005	32·1 1005	33·1 1005	34·1 1005
42·8 6 C.	23·6 1003	24·9 1003	26 1004	27·2 1001	28·4 1004	29·6 1004	30·6 1005	31·6 1005	32·6 1005	33·6 1005
44·6 7 C.	23·3 1002	24·6 1003	25·7 1003	26·9 1003	28 1003	29·2 1003	30·2 1004	31.2 1004	32·2 1001	33·2 1004
46·4 8 C.	23 1002	24·2 1002	25·3 1003	26·5 1003	27·6 1003	28·8 1003	29·8 1003	30·8 1003	31·8 1003	32·8 1003
48·2 9 C.	22·7 1002	23·9 1002	25 1002	26·1 1002	27·2 1002	28·4 1003	29·4 1003	30 4 1003	31·4 1003	32·4 1003
50·0 10 C.	22·4 1001	23·5 1002	24·6 1002	25·7 1002	26·8 1002	27·9 1002	29 1002	30 1002	31 1002	32 1002
51·8 11 C.	22·1 1001	23·2 1001	24·3 1001	25·4 1001	26·5 1002	27·6 1002	28·6 1002	29·6 1002	30·6 1002	31·6 1002
53·6 12 C.	21·8 1001	22·9 1001	24 1001	25·1 1001	26·1 1001	27·2 1001	28·2 1001	29·2 1001	30·2 1001	31·2 1001
55·4 13 C.	21·5 1001	22·6 1001	23·6 1001	24·7 1001	25·7 1001	26·8 1001	27·8 1001	28·8 1001	29·8 1001	30·8 1001
57·2 14 C.	21 2 1000	22·3 1000	23·3 1000	24·3 1000	25·3 1000	26·4 1000	27·4 1000	28·4 1000	29·4 1000	30·4 1000
59·0 15 C.	21 1001	22 1000	23 1000	24 1000	25 1000	26 1000	27 1000	28 1000	29 1000	30 1000
60·8 16 C.	20·7 1000	21·7 1000	22·7 1000	23·7 1000	24·7 1000	25·7 1000	26·6 1000	27·6 1000	28·6 1000	29·6 1000
62·6 17 C.	20·4 999	21·4 999	22·4 999	23·4 999	24·4 999	25·4 999	26·3 999	27·3 999	28·2 999	29·2 999
64·4 18 C.	20·1 999	21·1 999	22 999	23 999	24 999	25 999	25·9 999	26·9 999	27·8 999	28·8 999
66·2 19 C.	19·8 999	20·8 999	21 7 999	22 7 999	23·6 998	24·6 998	25·5 998	26·5 998	27·4 998	28·4 998
68·0 20 C.	19·5 999	20·5 998	21·4 998	22·4 998	23·3 998	24·3 998	25·2 998	26 1 998	27·1 998	28 998
69·8 21 C.	19·1 998	20·1 998	21.1 998	22·1 998	23 998	23·9 998	24·8 998	25·7 998	26·7 997	27·6 997
71·6 22 C.	18·8 998	19·8 998	20·7 998	21·7 997	22·6 997	23·6 997	24·4 997	25·3 997	26·3 997	27·2 997
73·4 23 C.	18·5 998	19·5 997	20·4 997	21·4 997	22·3 997	23 2 997	24·1 997	25 997	25·9 997	26·8 997
75·2 24 C.	18·3 997	19·2 997	20·1 997	21·1 997	21·9 997	22·8 997	23·7 997	24·6 996	25·5 996	26·4 996
77·0 25 C.	18 997	18·9 997	19·8 997	20·7 997	21·6 996	22·5 996	23·3 996	24·3 996	25·2 996	26·1 996
78·8 26 C.	17·7 997	18·6 996	19·5 996	20·4 996	21·3 996	22·2 996	23 996	23·9 996	24·8 995	25·7 995
80·6 27 C.	17·4 996	18·3 996	19·2 996	20·1 996	20·9 996	21·8 996	22·7 996	23·6 996	24·4 905	25·3 995
82·4 28 C.	17 996	18 996	18·9 996	19·7 995	20·6 995	21·5 995	22·3 995	23·2 995	24 995	24·9 994
84·2 29 C.	16·7 996	17·6 996	18·5 995	19·4 995	20·3 995	21·1 995	21·9 995	22·8 994	23·7 904	24·5 994
86·0 30 C.	16·4 995	17·3 995	18·2 995	19·1 995	19·9 995	20·8 904	21·6 994	22·5 994	23·3 994	24·2 994

Temp. Fahr.	Observed per centage of the Alcoholometer.									
	31 p cent.	32 p cent.	33 p.cent.	34 p.cent.	35 p.cent.	36 p.cent.	37 p.cent.	38 p.cent.	39 p.cent.	40 p.cent.
32·0° 0° C.	37·3 1009	38·3 1009	39·2 1009	40·2 1009	41·1 1009	42·1 1010	43·1 1010	44 1010	45 1010	45·9 1011
33·8 1 C.	36·8 1008	37·8 1008	38·8 1008	39·8 1008	40·8 1009	41·8 1009	42·7 1009	43·7 1009	44·6 1010	45 5 1010
35·6 2 C.	36·4 1007	37·4 1007	38·4 1008	39·4 1008	40·4 1008	41·4 1008	42·3 1008	43·3 1009	44·2 1009	45·1 1009
37·4 3 C.	36 1007	37 1007	38 1007	39 1007	40 1007	41 1008	42 1008	42·9 1008	43·9 1008	44·8 1008
39·2 4 C.	35·5 1006	36·5 1006	37·5 1006	38·5 1007	39·5 1007	40·5 1007	41·5 1007	42·5 1007	43·5 1007	44·4 1008
41·0 5 C	35·1 1005	36·1 1006	37·1 1006	38·1 1006	39·1 1006	40·1 1006	41·1 1007	42·1 1007	43·1 1007	44 1007
42·8 6 C	34·7 1005	35·7 1005	36·7 1005	37·7 1005	38·7 1005	39·7 1006	40·7 1006	41 1006	42·6 1006	43·6 1006
44·6 7 C.	34·2 1001	35·2 1001	36·2 1004	37·2 1005	38·2 1005	39·2 1005	40·2 1005	41·2 1005	42·2 1005	43·2 1005
46·4 8 C	33·8 1001	34·8 1004	35·8 1004	36·8 1004	37·8 1004	38·8 1004	39·8 1004	40·8 1004	41·8 1001	42·8 1005
48·2 9 C.	33·4 1003	34·4 1003	35·4 1003	36·4 1003	37·4 1004	38·4 1004	39·4 1004	40 4 1004	41·4 1001	42·4 1004
50·0 10 C.	33 1002	34 1002	35 1003	36 1003	37 1003	38 1003	39 1003	40 1003	41 1003	42 1003
51·8 11 C.	32·6 1002	33·6 1002	34·6 1002	35·6 1002	36·6 1002	37·6 1002	38·6 1002	39·6 1002	40·6 1003	41·6 1003
53·6 12 C.	32·2 1001	33·2 1001	34·2 1002	35·2 1002	36·2 1002	37·2 1002	38·2 1002	39·2 1002	40·2 1002	41·2 1002
55·4 13 C.	31·8 1001	32·8 1001	33·8 1001	34·8 1001	35·8 1001	36·8 1001	37·8 1001	38·8 1001	39·8 1001	40·8 1001
57·2 14 C.	31·4 1000	32·4 1000	33·4 1001	34·4 1001	35·4 1001	36·4 1001	37·4 1001	38·4 1001	39·4 1001	40·4 1001
59·0 15 C	31 1000	32 1000	33 1000	34 1000	35 1000	36 1000	37 1000	38 1000	39 1000	40 1000
60·8 16 C.	30·6 1000	31·6 1000	32.5 999	33·5 999	34·5 999	35·5 999	36·5 999	37·5 999	38·5 999	39·5 999
62·6 17 C.	30·2 999	31·2 999	32·1 999	33·1 999	34·1 999	35·1 999	36·1 999	37 1 999	38·1 999	39·1 999
64·4 18 C	29·8 999	30·8 999	31·7 998	32·7 998	33·7 998	34·7 998	35 7 998	36·7 998	37·7 998	38·7 998
66·2 19 C.	29·4 998	30·4 998	31·3 998	32·3 998	33·3 998	34·3 998	35·3 998	36·3 998	37·3 997	38·3 997
68·0 20 C	29 998	30 998	30·9 997	31·9 997	32·9 997	33·9 997	34·9 997	35·9 997	36·9 997	37·9 997
69·8 21 C.	28·6 997	29·6 997	30·5 997	31·5 997	32·5 997	33·5 997	34·5 997	35·5 996	36·5 996	37·5 996
71·6 22 C.	28·2 997	29·2 997	30·1 996	31·1 996	32·1 996	33·1 996	34·1 996	35·1 996	36·1 996	37·1 996
73·4 23 C.	27·8 996	28·8 996	29·7 996	30·7 996	31·7 996	32·7 996	33·7 996	34·7 995	35·7 995	36·7 995
75·2 24 C.	27·4 996	28·4 996	29·3 995	30·3 995	31·3 995	32·3 995	33·3 995	34·3 995	35·3 995	36·3 994
77·0 25 C.	27 995	28 995	28·9 995	29·9 995	30·9 995	31·9 994	32·9 994	33·9 994	34·9 994	35·9 994
78·8 26 C.	26·6 995	27·6 995	28·5 995	29·5 994	30·5 994	31·5 994	32·5 994	33·5 994	34·5 993	35·5 993
80·6 27 C.	26·2 995	27·2 994	28·1 994	29·1 994	30·1 994	31·1 993	32·1 993	33·1 993	34·1 993	35·1 993
82·4 28 C	25·8 994	26·8 994	27·7 994	28·7 993	29·7 993	30·7 993	31·7 993	32·7 993	33·7 992	34·7 992
84·2 29 C.	25·4 994	26·4 993	27·3 993	28·3 993	29·3 993	30·3 992	31·3 992	32·3 992	33·3 992	34·3 992
86·0 30 C.	25·1 993	26 993	26·9 993	27·9 993	28·9 992	29·9 992	30·9 992	31·9 991	32·9 991	33·9 991

(Gay-Lussac).—Table I.—Continued.

Temp Fahr.	Observed per centage of the Alcoholometer.										Temp. Fahr.	Observed per centage of the Alcoholometer.									
	41 p.cent.	42 p.cent	43 p.cent.	44 p.cent	45 p.cent.	46 p.cent.	47 p.cent.	48 p.cent.	49 p.cent.	50 p.cent.		51 p.cent.	52 p.cent.	53 p.cent.	54 p.cent.	55 p.cent.	56 p.cent.	57 p.cent.	58 p.cent.	59 p.cent.	60 p.cent.
32·0° 0° C.	46·9 1011	47·9 1011	48·8 1011	49·8 1011	50·7 1011	51·7 1011	52·6 1012	53·5 1012	54·5 1012	55·4 1012	32·0° 0° C.	56·1 1012	57·3 1012	58·3 1012	59·2 1012	60·2 1012	61·2 1012	62·1 1012	63.1 1013	64·1 1013	65 1013
33·8 1 C.	46·5 1010	47·5 1010	48.4 1010	49·4 1010	50·3 1010	51·3 1011	52·2 1011	53·2 1011	54·2 1011	55·1 1011	33·8 1 C.	56 1011	57 1011	57·9 1011	58·9 1011	59·9 1011	60·9 1011	61·8 1011	62·8 1012	63·8 1012	64·7 1012
35·6 2 C.	46·1 1009	47·1 1009	48·1 1009	49 1009	49·9 1010	50·9 1010	51·8 1010	52·8 1010	53·8 1010	54·7 1010	35·6 2 C.	55·7 1010	56·6 1010	57·6 1010	58·5 1010	59·5 1010	60·5 1011	61·5 1011	62·4 1011	63·4 1011	64·4 1011
37·1 3 C.	45·8 1008	46·7 1009	47·7 1009	48·6 1009	49·6 1009	50·5 1009	51·5 1009	52·4 1009	53·4 1009	54·3 1009	37·1 3 C.	55·3 1009	56·3 1009	57·2 1009	58·2 1010	59·2 1010	60·2 1010	61·1 1010	62·1 1010	63·1 1010	64·1 1010
39·2 4 C.	45·4 1008	46·4 1008	47·4 1008	48·3 1008	49·2 1008	50·2 1008	51·1 1008	52·1 1008	53 1008	54 1009	39·2 4 C.	55 1009	56 1009	56·9 1009	57·9 1009	58·9 1009	59·8 1009	60·8 1009	61·7 1009	62·7 1009	63·7 1009
41·0 5 C.	45 1007	45·9 1007	46·9 1007	47·9 1007	48·8 1007	49·8 1007	50·7 1007	51·7 1008	52·7 1008	53·6 1008	41·0 5 C.	54·6 1008	55·6 1008	56·6 1008	57·5 1008	58·5 1008	59·5 1008	60·4 1008	61·4 1008	62·4 1008	63·4 1008
42·8 6 C.	44·6 1006	45·5 1006	46·5 1006	47·5 1007	48·4 1007	49·4 1007	50·4 1007	51·4 1007	52·4 1007	53·3 1007	42·8 6 C.	54·3 1007	55·2 1007	56·2 1007	57·1 1007	58·1 1007	59·1 1007	60·1 1007	61 1008	62 1008	63 1008
44·6 7 C.	44·2 1005	45·1 1006	46·1 1006	47·1 1006	48·1 1006	49·1 1006	50·1 1006	51 1006	52 1006	52·9 1006	44·6 7 C.	53·9 1006	54·9 1006	55·9 1006	56·8 1006	57·8 1006	58·8 1006	59·8 1007	60·7 1007	61·7 1007	62·7 1007
46·4 8 C.	43·8 1005	44·8 1005	45·8 1005	46·8 1005	47·7 1005	48·7 1005	49·7 1005	50·6 1005	51·6 1005	52·6 1005	46·4 8 C.	53·6 1005	54·6 1005	55·5 1006	56·5 1006	57·5 1006	58·5 1006	59·5 1006	60·4 1006	61·4 1006	62·4 1006
48·2 9 C.	43·4 1004	44·4 1004	45·4 1004	46·4 1004	47·3 1004	48·3 1004	49·3 1005	50·2 1005	51·2 1005	52·2 1005	48·2 9 C.	53·2 1005	54·2 1005	55·1 1005	56·1 1005	57·1 1005	58·1 1005	59·1 1005	60 1005	61 1005	62 1005
50·0 10 C.	43 1003	44 1004	45 1004	46 1004	46·9 1004	47·9 1004	48·9 1004	49·9 1004	50·9 1004	51·8 1004	50·0 10 C.	52·8 1004	53·8 1004	54·8 1004	55·8 1004	56·8 1004	57·8 1004	58·8 1004	59·7 1004	60·7 1004	61·7 1004
51·8 11 C.	42·6 1003	43·6 1003	44·6 1003	45·6 1003	46·6 1003	47·6 1003	48·6 1003	49·5 1003	50·5 1003	51·5 1003	51·8 11 C.	52·5 1003	53·5 1003	54·4 1003	55·4 1003	56·4 1003	57·4 1003	58·4 1003	59·4 1003	60·4 1003	61·4 1003
53·6 12 C.	42·2 1002	43·2 1002	44·2 1002	45·2 1002	46·2 1002	47·2 1002	48·2 1002	49·2 1002	50·2 1002	51·1 1002	53·6 12 C.	52·1 1002	53·1 1002	54·1 1002	55 1002	56 1002	57 1002	58 1002	59 1002	60 1002	61 1002
55·4 13 C.	41·8 1001	42·8 1001	43·8 1001	44·8 1002	45·8 1002	46·8 1002	47·8 1002	48·8 1002	49·8 1002	50·8 1002	55·4 13 C.	51·8 1002	52·7 1002	53·7 1002	54·7 1002	55·7 1002	56·7 1002	57·7 1002	58·7 1002	59·7 1002	60·7 1002
57·2 14 C.	41·4 1001	42·4 1001	43·4 1001	44·4 1001	45·4 1001	46·4 1001	47·4 1001	48·4 1001	49·4 1001	50·4 1001	57·2 14 C.	51·4 1001	52·3 1001	53·3 1001	54·3 1001	55·3 1001	56·3 1001	57·3 1001	58·3 1001	59·3 1001	60·3 1001
59·0 15 C.	41 1000	42 1000	43 1000	44 1000	45 1000	46 1000	47 1000	48 1000	49 1000	50 1000	59·0 15 C.	51 1000	52 1000	53 1000	54 1000	55 1000	56 1000	57 1000	58 1000	59 1000	60 1000
60·8 16 C.	40·6 999	41·6 999	42·6 999	43·6 999	44·6 999	45·6 999	46·6 999	47·6 999	48·6 999	49·6 999	60·8 16 C.	50·6 999	51·6 999	52.6 999	53·6 999	54·6 999	55·6 999	56·6 999	57·6 999	58·6 999	59·6 999
62·6 17 C.	40·2 999	41·2 999	42·2 999	43·2 998	44·2 998	45·2 998	46·2 998	47·2 998	48·3 998	49·3 998	62·6 17 C.	50·3 998	51·3 998	52·3 998	53·3 998	54·3 998	55·3 998	56·3 998	57·3 998	58·3 998	59·3 998
64·4 18 C.	39·8 998	40·8 998	41·8 998	42·8 998	43·8 998	44·9 998	45·9 998	46·9 998	47·9 998	48·9 998	64·4 18 C.	49·9 998	50·9 998	51·9 998	52·9 998	53·9 998	54·9 998	55·9 998	56·9 997	57·9 997	58·9 997
66·2 19 C.	39·4 997	40·4 997	41·4 997	42·5 997	43·5 997	44·5 997	45·5 997	46·5 997	47·5 997	48·5 997	66·2 19 C.	49·5 997	50·6 997	51·6 997	52·6 997	53·6 997	54·6 997	55·6 997	56·6 997	57·6 997	58·6 997
68·0 20 C.	39 997	40 997	41 997	42·1 997	43·1 996	44·1 996	45·1 996	46·1 996	47·2 996	48·2 996	68·0 20 C.	49·2 996	50·2 996	51·2 996	52·2 996	53·2 996	54·2 996	55·2 996	56·2 996	57·2 996	58·2 996
69·8 21 C.	38·6 996	39·6 996	40.6 996	41·7 996	42·7 996	43·7 996	44·8 996	45·8 996	46·8 995	47·8 995	69·8 21 C.	48·8 995	49·8 995	50·8 995	51·8 995	52·9 995	53·9 995	54·9 995	55·9 995	56·9 995	57·9 995
71·6 22 C.	38·2 996	39·2 995	40·2 995	41·3 995	42·3 995	43·3 995	44·3 995	45·3 995	46·4 995	47·4 995	71·6 22 C.	48·4 995	49·4 995	50·4 995	51·4 994	52·5 994	53·5 994	54·5 994	55·5 994	56·5 994	57·5 994
73·4 23 C.	37·8 995	38·8 995	39·8 995	40·9 994	41·9 994	42·9 994	43·9 994	44·9 994	46 994	47 994	73·4 23 C.	48 994	49·1 994	50·1 994	51·1 994	52·1 994	53·1 994	54·1 993	55·1 993	56·1 993	57·1 993
75·2 24 C.	37·4 994	38·4 994	39·4 994	40·5 994	41·5 994	42·5 994	43·6 994	44·6 994	45·6 993	46·6 993	75·2 24 C.	47·6 993	48·7 993	49·7 993	50·7 993	51·8 993	52·8 993	53·8 993	54·8 993	55·8 993	56·8 992
77·0 25 C.	37 994	38 994	39 993	40·1 993	41·1 993	42·2 993	43·2 993	44·2 993	45·2 993	46·3 993	77·0 25 C.	47·3 993	48·3 993	49·3 993	50·3 992	51·4 992	52·4 992	53·4 992	54·4 992	55·5 992	56·5 992
78·8 26 C.	36·5 993	37·6 993	38·6 993	39·7 993	40·7 992	41·8 992	42·8 992	43·8 992	44·9 992	45·9 992	78·8 26 C.	46·9 992	47·9 992	49 992	50 991	51 991	52 991	53 991	54 991	55·1 991	56·1 991
80·6 27 C.	36·1 992	37·2 992	38·2 992	39·3 992	40·3 992	41·4 992	42·4 992	43·4 991	44·5 991	45·5 991	80·6 27 C.	46·5 991	47·6 991	48·6 991	49·6 991	50·7 990	51·7 990	52·7 990	53·7 990	54·8 990	55·8 990
82·4 28 C.	35·7 992	36·8 992	37·8 992	38·9 991	39·9 991	41 991	42 991	43 991	44·1 991	45·1 990	82·4 28 C.	46·1 990	47·2 990	48·2 990	49·2 990	50·3 990	51·3 990	52·3 990	53·3 989	54·4 989	55·4 989
84·2 29 C.	35·3 991	36·3 991	37·4 991	38·5 991	39·5 991	40·6 990	41·6 990	42·6 990	43·7 990	44·7 990	84·2 29 C.	45·7 990	46·8 989	47·8 989	48·9 989	49·9 989	51 989	52 989	53 989	54 989	55 988
86·0 30 C.	34·9 991	35·9 991	37 990	38·1 990	39·1 990	40·2 990	41·2 990	42·3 989	43·3 989	44·3 989	86·0 30 C.	45·4 989	46·4 989	47·5 989	48·5 988	49·6 988	50·6 988	51·6 988	52·6 988	53·6 988	54·7 988

(GAY LUSSAC).—TABLE I.—Continued.

Temp Fahr.	Observed per centage of the Alcoholometer.									
	61 p.cent.	62 p.cent.	63 p.cent.	64 p.cent.	65 p.cent.	66 p.cent.	67 p.cent.	68 p.cent.	69 p.cent.	70 p.cent.
32·0° 0° C.	66 1013	67 1013	68 1013	68·9 1013	69·9 1013	70·8 1013	71·8 1013	72·7 1013	73·7 1014	74·7 1014
33·8 1 C.	65·7 1012	66·7 1012	67·7 1012	68·6 1012	69·6 1012	70·5 1012	71·5 1012	72·4 1012	73·4 1013	74·3 1013
35·6 2 C.	65·3 1011	66·3 1011	67·3 1011	68·3 1011	69·3 1011	70·2 1011	71·2 1011	72·1 1012	73·1 1012	74 1012
37·4 3 C.	65 1010	66 1010	67 1010	68 1010	68·9 1010	69·9 1011	70·8 1011	71·8 1011	72·8 1011	73·7 1011
39·2 4 C.	64·7 1009	65·7 1009	66·6 1009	67·6 1010	68·6 1010	69·5 1010	70·5 1010	71·5 1010	72·5 1010	73·4 1010
41·0 5 C.	64·3 1009	65·3 1009	66·3 1009	67·3 1009	68·3 1009	69·2 1009	70·2 1009	71·2 1009	72·2 1009	73·1 1009
42·8 6 C.	64 1008	65 1008	66 1008	67 1008	68 1008	68·9 1008	69·9 1008	70·9 1008	71·9 1008	72·8 1008
44·6 7 C.	63·7 1007	64·7 1007	65·7 1007	66·7 1007	67·6 1007	68·6 1007	69·6 1007	70·6 1007	71·5 1007	72·5 1007
46·4 8 C.	63·4 1006	64·4 1006	65·4 1006	66·4 1006	67·3 1006	68·3 1006	69·3 1006	70·2 1006	71·2 1006	72·2 1006
48·2 9 C.	63 1005	64 1005	65 1005	66 1005	67 1005	67·9 1005	68·9 1005	69·9 1005	70·9 1005	71·9 1005
50·0 10 C.	62·7 1004	63·7 1004	64·7 1004	65·7 1004	66·7 1004	67·6 1004	68·6 1004	69·6 1004	70·6 1004	71·6 1004
51·8 11 C.	62·4 1003	63·4 1003	64·4 1003	65·4 1003	66·4 1003	67·3 1003	68·3 1003	69·3 1004	70·3 1004	71·3 1004
53·6 12 C.	62 1002	63 1002	64 1002	65 1002	66 1002	67 1002	68 1003	69 1003	70 1003	71 1003
55·4 13 C.	61·7 1002	62·7 1002	63·7 1002	64·7 1002	65·7 1002	66·7 1002	67·7 1002	68·7 1002	69·6 1002	70·6 1002
57·2 14 C.	61·3 1001	62·3 1001	63·3 1001	64·3 1001	65·3 1001	66·3 1001	67·3 1001	68·3 1001	69·3 1001	70·3 1001
59·0 15 C.	61 1000	62 1000	63 1000	64 1000	65 1000	66 1000	67 1000	68 1000	69 1000	70 1000
60·8 16 C.	60·6 999	61·7 999	62·7 999	63·7 999	64·7 999	65·7 999	66·7 999	67·7 999	68·7 999	69·7 999
62·6 17 C.	60·3 998	61·3 998	62·3 998	63·3 998	64·3 998	65·3 998	66·3 998	67·3 998	68·3 998	69·3 998
64·4 18 C.	59·9 997	61 997	62 997	63 997	64 997	65 997	66 997	67 997	68 997	69 997
66·2 19 C.	59·6 997	60·6 997	61·6 997	62·7 997	63·7 997	64·7 997	65·7 997	66·7 997	67·7 996	68·7 996
68·0 20 C.	59·2 996	60·3 996	61·3 996	62·3 996	63·3 996	64·3 996	65·4 996	66·4 996	67·4 996	68·4 996
69·8 21 C.	58·9 995	59·9 995	61 995	62 995	63 995	64 995	65 995	66 995	67 995	68·1 995
71·6 22 C.	58·5 994	59·5 994	60·6 994	61·6 994	62·7 994	63·7 994	64·7 994	65·7 994	66·7 994	67·8 994
73·4 23 C.	58·1 993	59·2 993	60·2 993	61·3 993	62·3 993	63·3 993	64·3 993	65·4 993	66·4 993	67·4 993
75·2 24 C.	57·8 992	58·9 992	59·9 992	61 992	62 992	63 992	64 992	65 992	66 992	67·1 992
77·0 25 C.	57·5 992	58·5 992	59·5 992	60·6 991	61·6 991	62·6 991	63·7 991	64·7 991	65·7 991	66·7 991
78·8 26 C.	57·1 991	58·1 991	59·2 991	60·2 991	61·3 990	62·3 990	63·3 990	64·3 990	65·3 990	66·4 990
80·6 27 C.	56·8 990	57·8 990	58·9 990	59·9 990	60·9 990	61·9 990	63 989	64 989	65 989	66 989
82·4 28 C.	56·4 989	57·5 989	58·5 989	59·5 989	60·6 989	61·6 989	62·6 989	63·7 989	64·7 989	65·7 988
84·2 29 C.	56 988	57·1 988	58·1 988	59·2 988	60·2 988	61·2 988	62·3 988	63·3 988	64·3 988	65·4 988
86·0 30 C.	55·7 988	56·7 987	57·8 987	58·8 987	59·9 987	60·9 987	61·9 987	63 987	64 987	65 987

Temp. Fahr.	Observed per centage of the Alcoholometer.									
	71 p.cent.	72 p.cent.	73 p.cent.	74 p.cent.	75 p.cent.	76 p.cent.	77 p.cent.	78 p.cent.	79 p.cent.	80 p.cent.
32·0° 0° C.	75·6 1014	76·6 1014	77·6 1014	78·6 1014	79·5 1014	80·5 1014	81·5 1014	82·4 1014	83·3 1014	84·3 1014
33·8 1 C.	75·3 1013	76·3 1013	77·3 1013	78·3 1013	79·2 1013	80·2 1013	81·2 1013	82·1 1013	83·1 1013	84 1013
35·6 2 C.	75 1012	76 1012	77 1012	78 1012	78·9 1012	79·9 1012	80·9 1012	81·9 1012	82·8 1012	83·7 1012
37·4 3 C.	74·7 1011	75·7 1011	76·7 1011	77·7 1011	78·6 1011	79·6 1011	80·6 1011	81·6 1011	82·5 1011	83·5 1011
39·2 4 C.	74·4 1010	75·3 1010	76·3 1010	77·3 1010	78·3 1010	79·3 1010	80·3 1010	81·3 1010	82·2 1010	83·2 1010
41·0 5 C.	74·1 1009	75 1009	76 1009	77 1009	78 1009	79 1009	80 1009	81 1009	81·9 1010	82·9 1010
42·8 6 C.	73·8 1008	74·7 1008	75·7 1008	76·7 1008	77·7 1008	78·7 1008	79·7 1008	80·7 1008	81·6 1008	82·6 1009
44·6 7 C.	73·5 1007	74·4 1007	75·4 1007	76·4 1007	77·4 1007	78·4 1007	79·4 1007	80·4 1007	81·4 1007	82·3 1008
46·4 8 C.	73·2 1006	74·1 1006	75·1 1006	76·1 1006	77·1 1006	78·1 1006	79·1 1007	80·1 1007	81·1 1007	82 1007
48·2 9 C.	72·9 1005	73·8 1005	74·8 1005	75·8 1005	76·8 1005	77·8 1005	78·8 1006	79·8 1006	80·8 1006	81·7 1006
50·0 10 C	72·6 1004	73·5 1004	74·5 1005	75·5 1005	76·5 1005	77·5 1005	78·5 1005	79·5 1005	80·5 1005	81·5 1005
51·8 11 C.	72·3 1004	73·2 1004	74·2 1004	75·2 1004	76·2 1004	77·2 1004	78·2 1004	79·2 1004	80·2 1004	81·2 1004
53·6 12 C.	72 1003	72·9 1003	73·9 1003	74·9 1003	75·9 1003	76·9 1003	77·9 1003	78·9 1003	79·9 1003	80·9 1003
55·4 13 C	71·6 1002	72·6 1002	73·6 1002	74·6 1002	75·6 1002	76·6 1002	77·6 1002	78·6 1002	79·6 1002	80·6 1002
57·2 14 C.	71·3 1001	72·3 1001	73·3 1001	74·3 1001	75·3 1001	76·3 1001	77·3 1001	78·3 1001	79·3 1001	80·3 1001
59·0 15 C	71 1000	72 1000	73 1000	74 1000	75 1000	76 1000	77 1000	78 1000	79 1000	80 1000
60·8 16 C.	70·7 999	71·7 999	72.7 999	73·7 999	74·7 999	75·7 999	76·7 999	77·7 999	78·7 999	79·7 999
62·6 17 C.	70·3 998	71·3 998	72·3 998	73·3 998	74·3 998	75·4 998	76·4 998	77·4 998	78·4 998	79·4 998
64·4 18 C.	70 997	71 997	72 997	73 997	74 997	75·1 997	76·1 997	77·1 997	78·1 997	79·1 997
66·2 19 C.	69·7 996	70·7 996	71·7 996	72·7 996	73·7 996	74·7 996	75·8 996	76·8 996	77·8 996	78·8 996
68·0 20 C.	69·4 996	70·4 996	71·4 995	72·4 995	73·4 995	74·4 995	75·5 995	76·5 995	77·5 995	78·5 995
69·8 21 C.	69·1 995	70·1 995	71·1 995	72·1 994	73·1 994	74·1 994	75·2 994	76·2 994	77·2 994	78·2 994
71·6 22 C.	68·8 994	69·8 994	70·8 994	71·8 994	72·8 993	73·8 993	74·8 993	75·9 993	76·9 993	77·9 993
73·4 23 C.	68·4 993	69·4 993	70·5 993	71·5 993	72·5 992	73·5 992	74·5 992	75·5 992	76·6 992	77·6 992
75·2 24 C	68·1 992	69·1 992	70·1 992	71·2 992	72·2 992	73·2 992	74·2 992	75·2 991	76·3 991	77·3 991
77·0 25 C.	67·8 991	68·8 991	69·8 991	70·8 991	71·8 991	72·8 991	73·9 991	74·9 991	76 991	77 991
78·8 26 C	67·4 990	68·4 990	69·5 990	70·5 990	71.5 990	72·5 990	73·6 990	74·6 990	75·6 990	76·7 990
80·6 27 C.	67·1 989	68·1 989	69·2 989	70·2 989	71·2 989	72·2 989	73·3 989	74·3 989	75·3 989	76·3 989
82·4 28 C	66·8 988	67·8 988	68·8 988	69·9 988	70·9 988	71·9 988	73 988	74 988	75 988	76 988
84·2 29 C.	66·4 988	67·4 987	68·5 987	69·5 987	70·6 987	71·6 987	72·6 987	73·7 987	74·7 987	75·7 987
86·0 30 C.	66·1 987	67·1 987	68·2 986	69·2 986	70·3 986	71·3 986	72·3 986	73·3 986	74·4 986	75·4 986

(GAY-LUSSAC).—TABLE I.—Concluded.

Temp Fahr.	Observed per centage of the Alcoholometer.									
	81 p.cent.	82 p.cent.	83 p.cent.	84 p.cent.	85 p.cent	86 p.cent.	87 p.cent.	88 p.cent.	89 p.cent.	90 p.cent.
32·0° 0° C.	85·2 1014	86·2 1014	87·1 1014	88 1014	88·9 1014	89·9 1015	90·8 1015	91·7 1015	92·6 1015	93·6 1015
33·8 1 C.	85 1013	85·9 1013	86·8 1013	87·8 1013	88·7 1013	89·6 1014	90·5 1014	91·5 1014	92·4 1014	93·3 1014
35·6 2 C.	84·7 1012	85·6 1012	86·6 1012	87·5 1012	88·5 1012	89·4 1013	90·3 1013	91·2 1013	92·2 1013	93·1 1013
37·4 3 C.	84·4 1011	85·4 1011	86·3 1011	87·3 1011	88·2 1011	89·2 1012	90·1 1012	91 1012	91·9 1012	92·9 1012
39·2 4 C.	84·2 1011	85·1 1011	86·1 1011	87 1011	87·9 1011	88·9 1011	89·8 1011	90·8 1011	91·7 1011	92·7 1011
41·0 5 C.	83·9 1010	84·8 1010	85·8 1010	86·7 1010	87·7 1010	88·6 1010	89·6 1010	90·5 1010	91·5 1010	92·4 1010
42·8 6 C.	83·6 1009	84·5 1009	85·5 1009	86·5 1009	87·4 1009	88·4 1009	89·3 1009	90·2 1009	91·2 1009	92·2 1009
44·6 7 C.	83·3 1008	84·2 1008	85·2 1008	86·2 1008	87·2 1008	88·1 1008	89·1 1008	90 1008	91 1008	91·9 1008
46·4 8 C.	83 1007	84 1007	85 1007	85·9 1007	86·9 1007	87·9 1007	88·8 1007	89·8 1007	90·7 1007	91·7 1007
48·2 9 C.	82·7 1006	83·7 1006	84·7 1006	85·7 1006	86·6 1006	87·6 1006	88·6 1006	89·5 1006	90·5 1006	91·5 1006
50·0 10 C.	82·4 1005	83·4 1005	84·4 1005	85·4 1005	86·4 1005	87·4 1005	88·3 1005	89·3 1005	90·2 1005	91·2 1005
51·8 11 C.	82·2 1004	83·1 1004	84·1 1004	85·1 1004	86·1 1004	87·1 1004	88 1004	89 1004	90 1004	91 1004
53·6 12 C.	81·9 1003	82·9 1003	83·9 1003	84·8 1003	85·8 1003	86·8 1003	87·8 1003	88·7 1003	89·7 1003	90·7 1003
55·4 13 C.	81·6 1002	82·6 1002	83·6 1002	84·6 1002	85·5 1002	86·5 1002	87·5 1002	88·5 1002	89·5 1002	90·5 1002
57·2 14 C.	81·3 1001	82·3 1001	83·3 1001	84·3 1001	85·3 1001	86·3 1001	87·3 1001	88·2 1001	89·2 1001	90·2 1001
59·0 15 C.	81 1000	82 1000	83 1000	84 1000	85 1000	86 1000	87 1000	88 1000	89 1000	90 1000
60·8 16 C.	80·7 999	81·7 999	82·7 999	83·7 999	84·7 999	85·7 999	86·7 999	87·7 999	88·7 999	89·7 999
62·6 17 C.	80·4 998	81·4 998	82·4 998	83·4 998	84·4 998	85·4 998	86·4 998	87·4 998	88·4 998	89·5 998
64·4 18 C.	80·1 997	81·1 997	82·1 997	83·1 997	84·1 997	85·2 997	86·2 997	87·2 997	88·2 997	89·2 997
66·2 19 C.	79·8 996	80·8 996	81·9 996	82·9 996	83·9 996	84·9 996	85·9 996	86·9 996	87·9 996	88·9 996
68·0 20 C.	79·5 995	80·5 995	81·6 995	82·6 995	83·6 995	84·6 995	85·6 995	86·6 995	87·7 995	88·7 995
69·8 21 C.	79·2 994	80·2 994	81·3 994	82·3 994	83·3 994	84·3 994	85·3 994	86·4 994	87·4 994	88·4 994
71·6 22 C.	78·9 993	79·9 993	81 993	82 993	83 993	84 993	85 993	86·1 993	87·1 993	88·2 993
73·4 23 C.	78·6 992	79·6 992	80·7 992	81·7 992	82·7 992	83·8 992	84·8 992	85·8 992	86·8 992	87·9 992
75·2 24 C	78·3 991	79·3 991	80·4 991	81·4 991	82·4 991	83·5 991	84·5 991	85·5 991	86·5 991	87·6 991
77·0 25 C.	78 991	79 991	80·1 990	81·1 990	82·1 990	83·2 990	84·2 990	85·2 990	86·3 990	87·4 990
78·8 26 C.	77·7 990	78·7 989	79·8 989	80·8 989	81·8 989	82·9 989	83·9 989	84·9 989	86 989	87·1 989
80·6 27 C.	77·4 989	78·4 988	79·5 988	80·5 988	81·5 988	82·6 988	83·6 988	84·7 988	85·7 988	86·8 988
82·4 28 C.	77·1 988	78·1 988	79·2 987	80·2 987	81·2 987	82·3 987	83·3 987	84·4 987	85·4 987	86·5 987
84·2 29 C.	76·7 987	77·8 987	78·9 987	79·9 986	80·9 986	82 986	83 986	84·1 986	85·1 986	86·2 986
86·0 30 C.	76·4 986	77·7 986	78·6 986	79·6 986	80·6 985	81·7 985	82·7 985	83·8 985	84·9 985	86 985

Temp. Fahr.	Observed per centage of the Alcoholometer.									
	91 p.cent.	92 p.cent.	93 p.cent.	94 p.cent.	95 p.cent.	96 p.cent.	97 p.cent.	98 p.cent.	99 p.cent.	100 p.cent.
32·0° 0° C.	94·5 1015	95·3 1015	96·2 1015	97·1 1015	98 1015	98·8 1015	99·7 1016			
33·8 1 C.	94·3 1014	95·1 1014	96 1014	96·9 1014	97·8 1014	98·6 1014	99·5 1014			
35·6 2 C.	94 1013	94·9 1013	95·8 1013	96·7 1013	97·6 1013	98·5 1013	99·3 1014			
37·4 8 C.	93·8 1012	94·7 1012	95·6 1012	96·5 1012	97·4 1012	98·3 1012	99·2 1012	1012		
39·2 4 C.	93·6 1011	94·5 1011	95·4 1011	96·3 1011	97·2 1011	98·1 1011	99 1011	99·9 1011		
41·0 5 C.	93·4 1010	94·3 1010	95·2 1010	96·1 1010	97 1010	97·9 1010	98·8 1010	99·7 1010		
42·8 6 C.	93·1 1009	94·1 1009	95 1009	95·9 1009	96·8 1009	97·8 1009	98·7 1009	99·6 1009		
44·6 7 C.	92·9 1008	93·9 1008	94·8 1008	95·7 1008	96·6 1008	97·6 1008	98·5 1008	99·4 1008		
46·4 8 C.	92·7 1007	93·6 1007	94·6 1007	95·5 1007	96·4 1007	97·4 1007	98·3 1007	99·2 1007	1007	
48·2 9 C.	92·5 1006	93·4 1006	94·4 1006	95·3 1006	96·2 1006	97·2 1006	98·1 1006	99·1 1006	100 1006	
50·0 10 C.	92·2 1005	93·2 1005	94·2 1005	95·1 1005	96 1005	97 1005	98 1005	98·9 1005	99·9 1005	
51·8 11 C.	92 1004	92·9 1004	93·9 1004	94·9 1004	95·8 1004	96·8 1004	97·8 1004	98·7 1004	99·7 1004	
53·6 12 C.	91·7 1003	92·7 1003	93·7 1003	94·7 1003	95·6 1003	96·6 1003	97·6 1003	98·5 1003	99·5 1003	
55·4 13 C.	91·5 1002	92·5 1002	93·5 1002	94·4 1002	95·4 1002	96·4 1002	97·4 1002	98·4 1002	99·3 1002	
57·2 14 C.	91·2 1001	92·2 1001	93·2 1001	94·2 1001	95·2 1001	96·2 1001	97·2 1001	98·2 1001	99·2 1001	
59·0 15 C.	91 1000	92 1000	93 1000	94 1000	95 1000	96 1000	97 1000	98 1000	99 1000	100 1000
60·8 16 C.	90·8 999	91·8 999	92.8 999	93·8 999	94·8 999	95·8 999	96·8 999	97·8 999	98·8 999	99·8 999
62·6 17 C.	90·5 998	91·5 998	92·6 998	93·6 998	94·6 998	95·6 998	96·6 998	97·6 998	98·7 998	99·7 998
64·4 18 C.	90·2 997	91·3 997	92·3 997	93·3 997	94·3 997	95·4 997	96·4 997	97·4 997	98·5 997	99·5 997
66·2 19 C.	90 996	91·1 996	92·1 996	93·1 996	94·1 996	95·2 996	96·2 996	97·3 996	98·3 996	99·3 996
68·0 20 C.	89·7 995	90·8 995	91·8 995	92·9 995	93·9 995	95 995	96 995	97·1 995	98·1 995	99·1 995
69·8 21 C.	89·5 994	90·5 994	91·6 994	92·6 994	93·7 994	94·7 994	95·8 994	96·9 994	97·9 994	99 994
71·6 22 C.	89·2 993	90·2 993	91·3 993	92·4 993	93·4 993	94·5 993	95·6 993	96·7 993	97·7 993	98·8 993
73·4 23 C.	89 992	90 992	91·1 992	92·1 992	93·2 992	94·3 992	95·4 992	96·5 992	97·5 992	98·6 992
75·2 24 C.	88·7 991	89·7 991	90·8 991	91·9 991	93 991	94·1 991	95·2 991	96·2 991	97·3 991	98·4 991
77·0 25 C.	88·4 990	89·5 990	90·6 990	91·6 990	92·7 990	93·8 990	94·9 990	96 990	97·1 990	98·2 990
78·8 26 C.	88·2 989	89·2 989	90·3 989	91·4 989	92.5 989	93·6 989	94·7 989	95·8 989	96·9 989	98·1 989
80·6 27 C.	87·9 988	89 988	90·1 988	91·1 988	92·2 988	93·4 988	94·5 988	95·6 987	96·7 987	97·9 987
82·4 28 C.	87·6 987	88·7 987	89·8 987	90·9 987	92 987	93·1 987	94·3 987	95·4 986	96·5 986	97·7 986
84·2 29 C.	87·3 986	88·4 986	89·5 986	90·6 986	91·7 986	92·9 986	94·1 986	95·2 986	96·3 985	97·5 985
86·0 30 C.	87·1 985	88·2 985	89·3 985	90·4 985	91·5 985	92·7 985	93·8 985	95 985	96·1 984	97·3 984

The second of GAY-LUSSAC'S tables corresponds with Table VII. of TRALLES, and gives directly, though less accurately, that which by the former table is only obtained by a calculation, namely, the percentage by volume of the liquid at any temperature at which it is tested, from the observed per cent. Thus, if, as in the former example, the alcoholometer indicated 59 per cent. in the liquid at 77° Fahr., the observed per cent., 59, is sought for in the upper horizontal column, and in the vertical column below it that number is then taken which is in the same horizontal column with the observed temperature, 77° Fahr., in the left-hand column, which in this case is 55, or the liquid at the observed temperature of 77° Fahr. contains 55 volumes of anhydrous alcohol.

(GAY-LUSSAC).—ALCOHOLOMETRIC TABLE II.

To find directly the percentage of absolute alcohol of a liquid at any temperature from the observed percentage at the same temperature.

Temp.		Observed per centage of the Alcoholometer.																			
Fahr.	Cent.	1 p.cent.	2 p.cent.	3 p.cent.	4 p.cent.	5 p.cent.	6 p.cent.	7 p.cent.	8 p.cent.	9 p.cent.	10 p.cent.	11 p.cent.	12 p.cent.	13 p.cent.	14 p.cent.	15 p.cent.	16 p.cent.	17 p.cent.	18 p.cent.	19 p.cent.	20 p.cent
32·0	0	1·3	2.4	3·4	4·1	5·4	6·5	7 5	8·6	9·7	10·9	12·2	13·4	14·7	16·1	17·5	19	20·4	21·7	23	24·3
33·3	1											—	13·4	14·7	16	17·3	18·7	20·1	21·4	22·7	24
35·6	2											—	13·4	14·7	16	17·2	18·6	19·9	21·2	22·4	23·7
37·4	3											—	13·3	14·6	15·9	17·1	18·3	19·7	20·9	22·1	23·4
39·2	4											—	13·3	14·5	15·8	16·9	18·1	19·4	20·7	21·9	23·1
41·0	5	1·4	2·5	3·5	4·5	5·5	6·6	7·7	8·7	9·8	10·9	12·1	13·2	14·4	15·7	16·8	18	19·2	20·5	21·6	22·8
42·8	6											—	13·1	14·3	15·6	16·7	17·8	19	20·3	21·4	22·5
44·6	7											—	13	14·2	15·4	16·6	17·7	18·8	20	21	22·1
46·4	8											—	13	14·1	15·3	16·4	17·5	18·6	19·7	20·7	21·8
48·2	9											—	12·9	14	15·1	16·2	17·3	18·4	19·5	20·5	21·6
50 0	10	1·4	2·4	3·4	4·5	5·5	6·5	7·5	8·5	9·5	10·6	11·7	12·7	13·8	14·9	16	17	18·1	19·2	20·2	21·3
51 8	11	1·3	2·4	3·4	4·4	5·4	6·4	7·4	8·4	9·4	10·5	11·6	12·6	13·6	14·7	15·8	16·8	17·9	19	20	21
53·6	12	1·2	2·3	3·3	4·3	5·3	6·3	7·3	8·3	9·3	10·4	11·5	12·5	13·5	14·6	15·6	16·6	17·6	18·7	19·7	20·7
55·4	13	1·2	2·2	3·2	4·2	5·2	6·2	7·2	8·2	9·2	10·3	11·4	12·4	13·4	14·4	15·4	16·4	17·4	18·5	19·5	20·5
57·2	14	1·1	2·1	3·1	4·1	5·1	6·1	7·1	8·1	9·1	10·2	11·2	12·2	13·2	14·2	15·2	16·2	17·2	18·2	19·2	20·2
59·0	15	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
60·8	16	0·9	1·9	2·9	3·9	4·9	5·9	6 9	7·9	8·9	9·9	10·9	11·9	12·9	13·9	14·9	15·9	16·9	17·8	18·7	19·7
62·6	17	0·8	1·8	2·8	3·8	4·8	5·8	6·8	7·8	8·8	9·8	10·8	11·7	12·7	13·7	14·7	15·6	16·6	17·5	18·4	19·4
64·4	18	0·7	1·7	2·7	3·7	4·7	5·7	6·7	7·7	8·7	9·7	10·7	11·6	12·5	13·5	14·5	15·4	16·3	17·3	18·2	19·1
66·2	19	0·6	1·6	2·6	3·6	4·5	5·5	6·5	7·5	8·5	9·5	10·5	11·4	12·4	13·3	14·3	15·2	16·1	17	17·9	18·8
68·0	20	0·5	1·5	2·4	3·4	4·4	5·4	6·4	7·3	8·3	9·3	10·3	11·2	12·2	13·1	14	14·9	15·8	16·7	17·6	18·5
69·8	21	0·4	1·4	2·3	3·3	4·3	5·2	6·2	7·1	8·1	9·1	10·1	11	11·9	12·8	13·7	14·6	15·5	16·4	17·3	18·2
71·6	22	0·3	1·3	2·2	3·2	4·1	5·1	6·1	7	7·9	8·9	9·9	10·8	11·7	12·6	13·5	14·4	15·3	16·2	17	17·9
73·4	23	0·1	1·1	2·1	3·1	4	4·9	5·9	6·8	7·8	8·7	9·7	10·6	11·5	12·4	13·3	14·1	15	15·9	16·7	17·6
75·2	24	0·0	1	1·9	2·9	3·8	4·8	5·8	6·7	7·6	8 5	9·5	10·4	11·3	12·2	13·1	13·9	14·8	15·7	16·5	17·4
77·0	25	—	0·8	1·7	2·7	3·6	4·6	5·5	6·5	7·4	8·3	9·3	10·2	11·1	12	12·8	13·6	14·5	15·4	16·2	17·1
78·8	26	—	0·7	1·6	2·6	3·5	4·4	5·4	6·3	7 2	8·1	9	9·9	10·8	11·7	12·6	13·4	14·2	15·1	15·9	16·7
80·6	27	—	0·5	1·5	2·4	3·3	4·3	5·2	6·1	7	7·9	8·8	9·7	10·6	11·5	12·3	13·1	13·9	14·8	15·6	16·4
82·4	28	—	0·3	1·3	2·2	3·1	4·1	5	5·9	6·8	7·7	8·6	9·5	10·3	11 2	12	12·8	13·6	14·4	15·2	16
84·2	29	—	0·1	1·1	2	2·9	3·9	4·8	5·7	6·6	7·5	8·4	9·2	10·1	11	11 7	12·5	13·3	14·1	14·9	15 7
86·0	30	—	0·0	0·9	1·9	2·8	3·7	4·6	5·5	6·4	7·3	8·1	9	9·8	10·7	11 5	12·3	13	13·8	14·6	15·4

Temp Fahr.	Cent.	21 p.cent.	23 p.cent.	23 p.cent.	24 p.cent.	25 p.cent.	26 p.cent.	27 p.cent.	28 p.cent.	29 p.cent.	30 p.cent.	31 p.cent.	32 p.cent.	33 p.cent.	34 p.cent.	35 p.cent.	36 p cent.	37 p cent.	38 p.cent.	39 p.cent.	40 p.cent.
32·0	0	25·7	27·1	28·5	29·9	31·1	32·3	33·4	34·5	35·6	36·6	37·6	38·5	39·6	40·6	41·5	42·5	43·5	44·4	45·4	46·4
33·8	1	25·4	26·8	28·1	29·4	30·6	31·8	32·9	34	35·1	36·1	37·1	38·1	39·1	40·1	41·2	42·2	43·1	44·1	45	46
35·6	2	25	26·4	27·6	28·9	30·2	31·4	32·5	33·5	34·6	35·6	36·7	37·7	38·7	39·7	40·7	41·7	42·7	43·7	44·6	45·5
37·4	3	24·7	26	27·3	28·6	29·8	31	32·1	33·1	34·1	35·2	36·2	37·3	38·3	39·3	40·3	41·3	42·3	43·2	44·2	45·2
39·2	4	24·4	25·7	26·9	28·1	29·3	30·6	31·6	32·7	33·7	34·7	35·7	36·7	37·7	38·8	39·8	40·8	41·8	42·8	43·8	44·8
41·0	5	24·1	25·3	26·5	27·7	28·9	30·1	31·2	32·3	33·3	34·3	35·3	36·3	37·3	38·3	39·3	40·3	41·4	42·4	43·4	44·4
42·8	6	23·7	25	26·1	27·3	28·5	29·7	30·8	31·8	32·8	33·8	34·9	35·9	36·9	37·9	38·9	39·9	40·9	41·9	42·9	43·9
44·6	7	23·4	24·7	25·8	27	28·1	29·3	30·3	31·3	32·3	33·3	34·3	35·4	36·4	37·4	38 4	39·4	40·4	41·4	42·4	43·4
46·4	8	23	24·2	25·4	26·6	27·7	28·9	29·9	30·9	31·9	32·9	33·9	34·9	35·9	36·9	38	39	40	41	42	43
48·2	9	22·7	23·9	25	26·2	27·3	28·5	29·5	30·5	31·5	32·5	33·5	34·5	35·5	36·5	37·5	38·6	39 6	40·6	41·6	42·6
50·0	10	22·4	23·5	24·6	25·8	26·9	28	29·1	30·1	31·1	32·1	33·1	34·1	35·1	36·1	37·1	38·1	39·1	40·1	41·1	42·1
51·8	11	22·1	23·2	24·3	25·4	26·5	27·7	28·7	29·7	30·7	31·7	32·7	33·7	34·7	35·7	36·7	37·7	38·7	39·7	40·7	41·7
53·6	12	21·8	22·9	24	25·1	26·1	27·2	28·2	29·2	30·2	31·2	32·2	33·2	34·3	35·3	36·3	37·3	38·3	39·3	40·3	41·3
55·4	13	21 5	22·6	23·7	24·7	25·7	26·8	27·8	28·8	29·8	30·8	31·8	32·8	33·8	34·8	35·8	36·8	37·8	38·8	39·8	40·9
57·2	14	21·2	22·3	23·3	24·3	25·3	26·4	27·4	28·4	29·4	30·4	31·4	32·4	33·4	34·4	35·4	36·4	37·4	38·4	39·4	40·4
59·0	15	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40
60·8	16	20·7	21·7	22·7	23·7	24·7	25·7	26·6	27·6	28·6	29·6	30·6	31·6	32·5	33·5	34·5	35·5	36·5	37·5	38·5	39·5
62·6	17	20·4	21·4	22·4	23·4	24·4	25·4	26·3	27·3	28·2	29·2	30·2	31·2	32·1	33·1	34·1	35·1	36·1	37·1	38·1	39·1
64·4	18	20·1	21·1	22	23	24	25	25·9	26·9	27·8	28·8	29·8	30·8	31·7	32·6	33.6	34·6	35·6	36·6	37·6	38·6
66·2	19	19·8	20·8	21·7	22·7	23·6	24·6	25·5	26·4	27·3	28·3	29·3	30·3	31·2	32·2	33·2	34·2	35·2	36·2	37·2	38·2
68·0	20	19·5	20·5	21·4	22·4	23·3	24·3	25·2	26·1	27	27·9	28·9	29·9	30·8	31·8	32·8	33·8	34·8	35·8	36·8	37·8
69·8	21	19·1	20·1	21·1	22·1	22·9	23·9	24·8	25·6	26·6	27·5	28·5	29·5	30·4	31·4	32·4	33·4	34·4	35·4	36·4	37·4
71·6	22	18·8	19·8	20·7	21·6	22·5	23·5	24·3	25·2	26·2	27·1	28·1	29·1	30	31	32	33	34	35	36	36·9
73·4	23	18·5	19·4	20·3	21·3	22·2	23·1	24	24·9	25·8	26·7	27·7	28·7	29·6	30·6	31·6	32·6	33·5	34·5	35·5	36·5
75·2	24	18·2	19·1	20	21	21·8	22·7	23·6	24·5	25·4	26·3	27·3	28·3	29·2	30·2	31·1	32·1	33·1	34·1	35·1	36·1

(Gay-Lussac).—Table II.—Continued.

Temp.		Observed per centage of the Alcoholometer.																			
Fahr.	Cent.	21 p.cent.	22 p.cent.	23 p.cent.	24 p.cent.	25 p.cent.	26 p.cent.	27 p.cent.	28 p.cent.	29 p.cent.	30 p.cent.	31 p.cent.	32 p.cent.	33 p.cent.	34 p.cent.	35 p.cent.	36 p.cent.	37 p.cent.	38 p.cent.	39 p.cent.	40 p.cent.
77·0	25	17·9	18·8	19·7	20·6	21·5	22·4	23·2	24·2	25·1	26	26·9	27·9	28·8	29·7	30·7	31·7	32·7	33·7	34·7	35·7
78·8	26	17·6	18·5	19·4	20·3	21·2	22·1	22·9	23·8	24·7	25·6	26·5	27·5	28·4	29·3	30·3	31·3	32·3	33·3	34·3	35·3
80·6	27	17·3	18·2	19·1	20	20·8	21·7	22·6	23·5	24·3	25·2	26·1	27·1	27·9	28·9	29·9	30·9	31·9	32·9	33·9	34·8
82·4	28	16·9	17·9	18·8	19·6	20·5	21·4	22·2	23·1	23·9	24·8	25·7	26·6	27·5	28·5	29·5	30·5	31·5	32·5	33·5	34·4
84·2	29	16·6	17·5	18·4	19·3	20·2	21	21·8	22·7	23·6	24·4	25·2	26·2	27·1	28·1	29·1	30·1	31·1	32·1	33·1	34
86·0	30	16·3	17·2	18·1	19	19·8	20·7	21·5	22·4	23·2	24	24·9	25·8	26·7	27·7	28·7	29·7	30·7	31·6	32·6	33·6

Temp. Fahr.	Temp. Cent.	41 p.cent.	42 p.cent.	43 p.cent.	44 p.cent.	45 p.cent.	46 p.cent.	47 p.cent.	48 p.cent.	49 p.cent.	50 p.cent.	51 p.cent.	52 p.cent.	53 p.cent.	54 p.cent.	55 p.cent.	56 p.cent.	57 p.cent.	58 p.cent.	59 p.cent.	60 p.cent.
32·0	0	47·4	48·4	49·3	50·3	51·3	52·3	53·2	54·1	55·1	56·1	57·1	58	59	59·9	60·9	61·9	62·9	63·9	64·9	65·8
33·8	1	47	48	48·9	49·9	50·8	51·8	52·8	53·7	54·7	55·7	56·7	57·6	58·6	59·6	60·6	61·6	62·5	63·5	64·5	65·5
35·6	2	46·5	47·5	48·5	49·5	50·1	51·4	52·3	53·3	54·3	55·3	56·3	57·2	58·2	59·2	60·2	61·2	62·1	63·1	64·1	65·1
37·4	3	46·2	47·1	48·1	49	50	51	52	52·9	53·9	54·8	55·8	56·8	57·8	58·8	59·8	60·8	61·7	62·7	63·7	64·7
39·2	4	45·8	46·7	47·7	48·7	49·6	50·6	51·5	52·5	53·5	54·5	55·5	56·5	57·4	58·4	59·4	60·3	61·3	62·3	63·3	64·3
41·0	5	45·3	46·2	47·2	48·2	49·2	50·2	51·1	52·1	53·1	54	55	56	57	58	59	60	60·9	61·9	62·9	63·9
42·8	6	44·9	45·8	46·8	47·8	48·8	49·8	50·8	51·7	52·7	53·7	54·7	55·6	56·6	57·5	58·5	59·5	60·5	61·5	62·5	63·5
44·6	7	44·4	45·4	46·4	47·4	48·4	49·4	50·4	51·3	52·3	53·2	54·2	55·2	56·2	57·1	58·1	59·1	60·1	61·1	62·1	63·1
46·4	8	44	45	46	47	47·9	48·9	49·9	50·9	51·9	52·9	53·9	54·9	55·8	56·8	57·8	58·8	59·8	60·8	61·8	62·8
48·2	9	43·6	44·6	45·6	46·6	47·5	48·5	49·5	50·5	51·5	52·5	53·5	54·5	55·4	56·4	57·4	58·4	59·4	60·4	61·4	62·4
50·0	10	43·1	44·1	45·1	46·1	47·1	48·1	49·1	50·1	51·1	52	53	54	55	56	57	58	59	60	61	62
51·8	11	42·7	43·7	44·7	45·7	46·7	47·7	48·7	49·7	50·7	51·7	52·7	53·7	54·6	55·6	56·6	57·6	58·6	59·6	60·6	61·6
53·6	12	42·3	43·3	44·3	45·3	46·3	47·3	48·3	49·3	50·3	51·2	52·2	53·2	54·2	55·2	56·2	57·2	58·2	59·2	60·2	61·2
55·4	13	41·9	42·9	43·9	44·9	45·9	46·9	47·9	48·9	49·9	50·9	51·9	52·8	53·8	54·8	55·8	56·8	57·8	58·8	59·8	60·8
57·2	14	41·4	42·4	43·4	44·4	45·4	46·4	47·4	48·4	49·4	50·4	51·4	52·4	53·4	54·4	55·4	56·4	57·4	58·4	59·4	60·4
59·0	15	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60
60·8	16	40·6	41·6	42·6	43·6	44·6	45·6	46·6	47·6	48·6	49·6	50·6	51·6	52·6	53·6	54·6	55·6	56·6	57·6	58·6	59·6
62·6	17	40·1	41·1	42·1	43·1	44·1	45·2	46·2	47·2	48·2	49·2	50·2	51·2	52·2	53·2	54·2	55·2	56·2	57·2	58·2	59·2
64·4	18	39·7	40·7	41·7	42·7	43·7	44·8	45·8	46·8	47·8	48·8	49·8	50·8	51·8	52·8	53·8	54·8	55·8	56·8	57·8	58·8
66·2	19	39·3	40·3	41·3	42·4	43·4	44·4	45·4	46·4	47·4	48·4	49·4	50·4	51·4	52·4	53·4	54·4	55·4	56·4	57·4	58·4
68·0	20	38·9	39·9	40·9	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58
69·8	21	38·4	39·4	40·4	41·5	42·5	43·5	44·6	45·6	46·6	47·6	48·6	49·6	50·6	51·6	52·6	53·6	54·6	55·6	56·6	57·6
71·6	22	38	39	40	41·1	42·1	43·1	44·1	45·1	46·1	47·1	48·1	49·1	50·1	51·1	52·2	53·2	54·2	55·2	56·2	57·2
73·4	23	37·6	38·6	39·6	40·6	41·6	42·6	43·6	44·6	45·7	46·7	47·7	48·8	49·8	50·8	51·8	52·8	53·8	54·8	55·8	56·8
75·2	24	37·2	38·2	39·2	40·2	41·2	42·2	43·3	44·3	45·3	46·3	47·3	48·4	49·4	50·4	51·4	52·4	53·4	54·4	55·4	56·4
77·0	25	36·7	37·7	38·7	39·8	40·8	41·9	42·9	43·9	44·9	46	47	48	49	50	51	52	53	54	55	56
78·8	26	36·3	37·3	38·3	39·4	40·4	41·5	42·5	43·5	44·5	45·5	46·5	47·5	48·5	49·5	50·5	51·5	52·5	53·5	54·5	55·6
80·6	27	35·9	36·9	37·9	39	40	41·1	42·1	43·1	44·1	45·1	46·1	47·1	48·1	49·1	50·2	51·2	52·2	53·2	54·2	55·2
82·4	28	35·4	36·5	37·5	38·6	39·6	40·6	41·6	42·6	43·7	44·7	45·7	46·7	47·7	48·7	49·8	50·8	51·8	52·8	53·8	54·8
84·2	29	35	36	37·1	38·1	39·1	40·2	41·2	42·2	43·3	44·3	45·3	46·3	47·3	48·4	49·4	50·4	51·4	52·4	53·4	54·4
86·0	30	34·6	35·6	36·6	37·7	38·7	39·8	40·8	41·8	42·8	43.8	44·9	45·9	47	48	49	50	51	52	53	54

Temp. Fahr.	Temp. Cent.	61 p.cent.	62 p.cent.	63 p.cent.	64 p.cent.	65 p.cent.	66 p.cent.	67 p.cent.	68 p.cent.	69 p.cent.	70 p.cent.	71 p.cent.	72 p.cent.	73 p.cent.	74 p.cent.	75 p.cent.	76 p.cent.	77 p.cent.	78 p.cent.	79 p.cent.	80 p.cent.
32·0	0	66·8	67·8	68·8	69·8	70·8	71·7	72·7	73·7	74·7	75·7	76·6	77·6	78·6	79·6	80·6	81·6	82·6	83·6	84·5	85·5
33·8	1	66·5	67·5	68·5	69·4	70·4	71·3	72·3	73·3	74·3	75·3	76·2	77·2	78·2	79·2	80·2	81·2	82·2	83·2	84·2	85·1
35·6	2	66·1	67·1	68·1	69·1	70·1	71	71·9	72·9	73·9	74·9	75·9	76·9	77·9	78·9	79·9	80·9	81·9	82·9	83·8	84·7
37·4	3	65·6	66·6	67·6	68·6	69·6	70·6	71·6	72·6	73·6	74·5	75·5	76·5	77·5	78·5	79·5	80·5	81·5	82·5	83·4	84·4
39·2	4	65·3	66·3	67·3	68·3	69·3	70·2	71·2	72·2	73·2	74·1	75·1	76·1	77·1	78·1	79·1	80·1	81·1	82·1	83	84
41·0	5	64·9	65·9	66·9	67·9	68·9	69·8	70·8	71·8	72·8	73·8	74·8	75·7	76·7	77·7	78·7	79·7	80·7	81·7	82·7	83·7
42·8	6	64·5	65·5	66·5	67·5	68·5	69·5	70·5	71·5	72·5	73·4	74·4	75·3	76·3	77·3	78·3	79·3	80·3	81·3	82·3	83·3
44·6	7	64·1	65·1	66·1	67·1	68·1	69·1	70·1	71·1	72	73	74	75	76	77	78	79	80	81	82	82·9
46·4	8	63·8	64·8	65·8	66·8	67·7	68·7	69·7	70·6	71·6	72·6	73·6	74·6	75·6	76·6	77·6	78·6	79·6	80·6	81·6	82·6
48·2	9	63·4	64·4	65·4	66·4	67·3	68·3	69·3	70·3	71·3	72·3	73·3	74·2	75·2	76·2	77·2	78·2	79·2	80·2	81·2	82·2
50·0	10	63	64	65	66	67	67·9	68·9	69·9	70·9	71·9	72·9	73·9	74·9	75·9	76·9	77·9	78·9	79·9	80·9	81·9
51·8	11	62·6	63·6	64·6	65·6	66·6	67·6	68·6	69·6	70·6	71·6	72·6	73·5	74·5	75·5	76·5	77·5	78·5	79·5	80·5	81·5
53·6	12	62·2	63·2	64·2	65·2	66·2	67·2	68·2	69·2	70·2	71·2	72·2	73·1	74·1	75·1	76·1	77·1	78·1	79·1	80·1	81·1
55·4	13	61·8	62·8	63·8	64·8	65·8	66·8	67·8	68·8	69·8	70·8	71·8	72·8	73·8	74·8	75·8	76·8	77·8	78·8	79·8	80·8
57·2	14	61·4	62·4	63·4	64·4	65·4	66·4	67·4	68·4	69·4	70·4	71·4	72·4	73·4	74·4	75·4	76·4	77·4	78·4	79·4	80·4
59·0	15	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80
60·8	16	60·6	61·6	62·6	63·6	64·6	65·6	66·6	67·6	68·6	69·6	70·6	71·6	72·6	73·6	74·6	75·6	76·6	77·6	78·6	79·6
62·6	17	60·2	61·2	62·2	63·2	64·2	65·2	66·3	67·2	68·2	69·2	70·2	71·2	72·2	73·2	74·2	75·2	76·2	77·2	78·2	79·2
64·4	18	59·8	60·8	61·8	62·8	63·8	64·8	65·8	66·8	67·8	68·8	69·8	70·8	71·8	72·8	73·8	74·9	75·9	76·9	77·9	78·9
66·2	19	59·4	60·4	61·4	62·5	63·5	64·5	65·5	66·5	67·5	68·5	69·5	70·5	71·5	72·5	73·5	74·5	75·5	76·5	77·5	78·5
68·0	20	59	60	61	62	63	64	65·1	66·1	67·1	68·1	69·1	70·1	71·1	72·1	73·1	74·1	75·1	76·1	77·1	78·1
69·8	21	58·6	59·6	60·7	61·7	62·7	63·7	64·7	65·7	66·7	67·7	68·7	69·7	70·7	71·7	72·7	73·7	74·7	75·8	76·8	77·8
71·6	22	58·2	59·2	60·3	61·3	62·3	63·3	64·3	65·3	66·3	67·3	68·3	69·3	70·3	71·3	72·3	73·3	74·3	75·4	76·4	77·4
73·4	23	57·8	58·8	59·8	60·9	61·9	62·9	63·9	64·9	65·9	66·9	67·9	68·9	70	71	72	73	74	75	76	77
75·2	24	57·4	58·4	59·4	60·5	61·5	62·5	63·5	64·5	65·5	66·5	67·5	68·5	69·6	70·6	71·6	72·6	73·6	74·6	75·6	76·6
77·0	25	57	58	59	60·1	61·1	62·1	63·1	64·1	65·1	66·1	67·1	68·1	69·2	70·2	71·2	72·2	73·2	74·2	75·3	76·3
78·8	26	56·6	57·6	58·6	59·6	60·7	61·7	62·7	63·7	64·7	65·7	66·7	67·7	68·8	69·8	70·8	71·8	72·8	73·8	74·8	75·9
80·6	27	56·2	57·2	58·3	59·3	60·3	61·3	62·3	63·3	64·3	65·3	66·3	67·3	68·4	69·4	70·4	71·4	72·4	73·4	74·4	75·5
82·4	28	55·8	56·8	57·8	58·8	59·9	60·9	61·9	62·9	63·9	64·9	66	67	68	69·1	70·1	71·1	72·1	73·1	74·1	75·1
84·2	29	55·4	56·4	57·4	58·5	59·5	60·5	61·5	62·5	63·5	64·5	65·6	66·6	67·7	68·7	69·7	70·7	71·7	72·7	73·7	74·7
86·0	30	55	56	57·1	58·1	59·1	60·1	61·1	62·1	63·1	64·1	65·2	66·2	67·3	68·3	69·3	70·3	71·3	72·3	73·3	74·3

(GAY-LUSSAC).—TABLE II.—Concluded.

Temp. Fahr.	Temp. Cent.	81 p.cent.	82 p.cent.	83 p.cent.	84 p.cent.	85 p.cent.	86 p.cent.	87 p.cent.	88 p.cent.	89 p.cent.	90 p.cent.	91 p.cent.	92 p.cent.	93 p.cent.	94 p.cent.	95 p.cent.	96 p.cent.	97 p.cent.	98 p.cent.	99 p.cent.	100 p.cent.
		Observed per centage of the Alcoholometer.																			
32·0	0	86·4	87·4	88·3	89·2	90·2	91·2	92·2	93·1	94	95	95·9	96·8	97·7	98·6	99·5	100·3	101·2			
33·8	1	86·1	87	88	89	89·9	90·8	91·8	92·8	93·7	94·6	95·6	96·5	97·4	98·3	99·2	100	100·9			
35·6	2	85·7	86·6	87·6	88·6	89·6	90·5	91·5	92·4	93·4	94·3	95·2	96·1	97	97·9	98·9	99·8	100·7			
37·4	3	85·3	86·3	87·3	88·3	89·2	90·2	91·2	92·1	93	94	94·9	95·8	96·7	97·7	98·6	99·5	100·4			
39·2	4	85	86	87	88	88·9	89·9	90·8	91·8	92·7	93·7	94·6	95·5	96·4	97·4	98·3	99·2	100·1	101		
41·0	5	84·7	85·6	86·6	87·6	88·5	89·5	90·5	91·4	92·4	93·3	94·3	95·2	96·2	97·1	98	98·9	99·8	100·7		
42·8	6	84·3	85·3	86·3	87·3	88·2	89·2	90·1	91	92	93	93·9	94·9	95·9	96·8	97·7	98·7	99·6	100·5		
44·6	7	83·9	84·9	85·9	86·9	87·9	88·8	89·8	90·7	91·7	92·6	93·6	94·6	95·6	96·5	97·4	98·4	99·3	100·2		
46·4	8	83·6	84·6	85·6	86·5	87·5	88·5	89·4	90·4	91·3	92·3	93·3	94·3	95·3	96·2	97·1	98·1	99	99·9		
48·2	9	83·2	84·2	85·2	86·2	87·1	88·1	89·1	90	91	92	93	94	95	95·9	96·8	97·8	98·7	99·7	100	
50·0	10	82·8	83·8	84·8	85·8	86·8	87·8	88·7	89·7	90·7	91·7	92·7	93·7	94·7	95·6	96·5	97·5	98·5	99·4	100·4	
51·8	11	82·5	83·4	84·4	85·4	86·4	87·4	88·4	89·4	90·4	91·4	92·4	93·3	94·3	95·3	96·2	97·2	98·2	99·1	100·1	
53·6	12	82·1	83·1	84·1	85	86	87	88	89	90	91	92	93	94	95	95·9	96·9	97·9	98·8	99·8	
55·4	13	81·8	82·8	83·8	84·8	85·7	86·7	87·7	88·7	89·7	90·7	91·7	92·7	93·7	94·6	95·6	96·6	97·6	98·6	99·5	
57·2	14	81·4	82·4	83·4	84·4	85·4	86·4	87·4	88·3	89·3	90·3	91·3	92·3	93·3	94·3	95·3	96·3	97·3	98·3	99·3	
59·0	15	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100
60·8	16	80·6	81·6	82·6	83·6	84·6	85·6	86·6	87·6	88·6	89·6	90·7	91·7	92·7	93·7	94·7	95·7	96·7	97·7	98·7	99·7
62·6	17	80·2	81·2	82·2	83·2	84·2	85·2	86·2	87·2	88·2	89·3	90·3	91·3	92·4	93·4	94·4	95·4	96·4	97·4	98·5	99·5
64·4	18	79·9	80·9	81·9	82·9	83·9	84·9	85·9	86·9	87·9	88·9	89·9	91	92	93	94	95·1	96·1	97·1	98·2	99·2
66·2	19	79·5	80·5	81·6	82·6	83·6	84·6	85·6	86·6	87·6	88·6	89·6	90·7	91·7	92·7	93·7	94·8	95·8	96·9	97·9	98·9
68·0	20	79·1	80·1	81·2	82·2	83·2	84·2	85·2	86·2	87·2	88·2	89·2	90·3	91·3	92·4	93·4	94·5	95·5	96·6	97·6	98·6
69·8	21	78·7	79·7	80·8	81·8	82·8	83·8	84·8	85·9	86·9	87·9	88·9	90	91	92	93·1	94·1	95·2	96·3	97·3	98·4
71·6	22	78·4	79·4	80·4	81·4	82·4	83·4	84·4	85·5	86·5	87·6	88·6	89·6	90·7	91·8	92·8	93·9	94·9	96	97	98·1
73·4	23	78	79	80·1	81·1	82·1	83·1	84·1	85·1	86·1	87·2	88·3	89·3	90·4	91·4	92·4	93·5	94·6	95·7	96·7	97·8
75·2	24	77·6	78·6	79·7	80·7	81·7	82·7	83·7	84·7	85·7	86·8	87·9	88·9	90	91·1	92·1	93·2	94·3	95·3	96·4	97·5
77·0	25	77·3	78·3	79·3	80·3	81·3	82·3	83·4	84·4	85·4	86·5	87·5	88·6	89·7	90·7	91·8	92·9	93·9	95	96·1	97·2
78·8	26	76·9	77·9	78·9	79·9	80·9	81·9	82·9	84	85	86·1	87·2	88·2	89·3	90·4	91·5	92·5	93·6	94·7	95·8	97
80·6	27	76·5	77·5	78·5	79·5	80·5	81·6	82·6	83·6	84·7	85·7	86·8	87·9	89	90	91·1	92·2	93·3	94·4	95·5	96·7
82·4	28	76·1	77·1	78·2	79·2	80·2	81·3	82·3	83·3	84·3	85·4	86·5	87·5	88·6	89·7	90·8	91·9	93	94·1	95·2	96·4
84·2	29	75·7	76·8	77·8	78·8	79·8	80·9	81·6	83	84	85	86·1	87·2	88·2	89·3	90·4	91·6	92·7	93·8	94·9	96·1
86·0	30	75·3	76·4	77·4	78·4	79·4	80·5	81·5	82·6	83·6	84·7	85·8	86·9	87·9	89	90·1	91·2	92·4	93·5	94·6	95·8

TRALLES' and GAY-LUSSAC'S alcoholometers have been very generally adopted in different countries. They both give the per cent. of alcohol by volume. If it be desired to know the per cent. by weight, it may be ascertained from the per cent. in volume of the liquid at 60°, by the following table :—

TABLE OF COMPARISON

Between the per cent. of alcohol by volume at 60° Fahr.—TRALLES'—and per cent. by weight.

Per cent. By volume.	Per cent. By weight.	Per cent. By weight.	Per cent. By volume.
0	0·	0	0·
5	4·00	5	6·25
10	8·05	10	12·42
15	12·15	15	18·52
20	16·28	20	24·57
25	20·46	25	30·55
30	24·69	30	36·45
35	28·99	35	42·25
40	33·39	40	47·92
45	37·90	45	53·13
50	42·52	50	58·79
55	47·29	55	63·97
60	52·20	60	68·97
65	57·25	65	73·79
70	62·51	70	78·40
75	67·93	75	82·80
80	73·59	80	86·97
85	79·50	85	90·88
90	85·75	90	94·16
95	92·46	95	97·61
100	100·00	100	100·00

Knowing the percentage volume of alcohol in a liquid at any temperature, the same results are arrived at when such percentage is multiplied by the specific gravity of the pure anhydrous spirit at the normal thermometric degree—0·7939 in TRALLES' tables, and 0·7947 in GAY-LUSSAC'S—and dividing this product by the density of the liquid at the observed temperature.

TABLE BY LOWITZ,

Giving the per cent. of absolute alcohol by weight, from the specific gravity at 68° Fahr. (20° C.)

Per cent. of alcohol by weight.	Specific gravity at 68°.	Per cent. of alcohol by weight.	Specific gravity at 68°.	Per cent. of alcohol by weight.	Specific gravity at 68°.	Per cent. of alcohol by weight.	Specific gravity at 68°.
100	791	74	859	48	919	23	968
99	794	73	861	47	921	22	970
98	797	72	863	46	923	21	971
97	800	71	866	45	925	20	973
96	803	70	868	44	927	19	974
95	805	69	870	43	930	18	976
94	808	68	872	42	932	17	977
93	811	67	875	41	934	16	978
92	813	66	877	40	936	15	980
91	816	65	880	39	938	14	981
90	818	64	882	38	940	13	983
89	821	63	885	37	942	12	985
88	823	62	887	36	944	11	986
87	826	61	889	35	946	10	987
86	828	60	892	34	948	9	988
85	831	59	894	33	950	8	989
84	834	58	896	32	952	7	991
83	836	57	899	31	954	6	992
82	839	56	901	30	956	5	994
81	842	55	903	29	957	4	995
80	844	54	905	28	959	3	997
79	847	53	907	27	961	2	998
78	849	52	909	26	963	1	999
77	851	51	912	25	965	0	1000
76	853	50	914	24	966		
75	856	49	917				

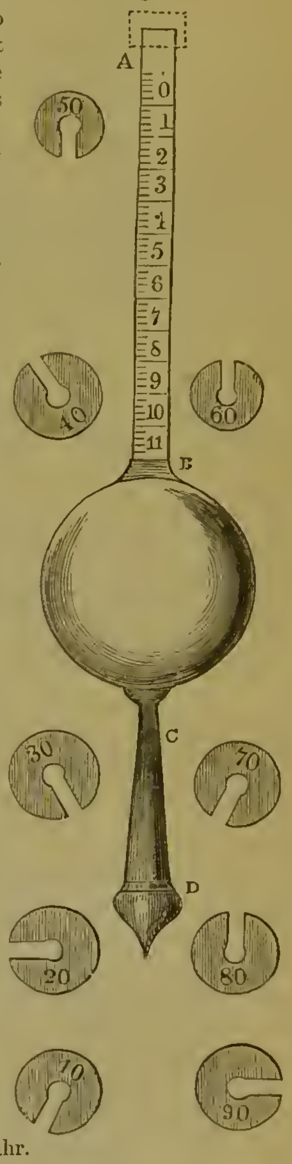
When a scale of per cent. by weight is added to TRALLES' alcoholometer, it sometimes bears the name of RICHTER'S scale.

In England, the amount of revenue which flows into the treasury annually from the trade in spirituous liquors, renders it a matter of importance to ascertain the real strength or percentage of alcohol with expedition and accuracy. Various forms of alcoholometers have therefore been constructed, some of which (Sikes' hydrometer for instance) have been sanctioned by the excise board. These instruments do not note the specific gravity of the liquid, but the excess or deficiency of alcohol above or below a standard liquor called *proof spirit*—a term which originated in a rude method of ascertaining the strength of spirit, by pouring it upon some gunpowder in a dish, and igniting it. This was called the proof. If the gunpowder took fire at the end of the combustion, the liquor was said to be above or over proof; but if the powder did not take fire the liquor was reputed to be below or under proof. The gunpowder test, however, is quite uncertain as to the quantity of spirit present in the liquid; for though the powder may be ignited when only a small quantity of spirit is used, yet on employing a larger amount, so much water may be left behind as to prevent the ignition. By parliamentary enactments the strength of proof spirit has been fixed at such a density that 13 volumes at 51° Fahr. should equal in weight 12 volumes of water at the same temperature. According to this standard, proof spirit has a gravity of ·9186 at 60° Fahr., and contains 57·27 per cent. by volume, or 49·50 per cent. by weight, of absolute alcohol. The liquors are estimated at the quantity of spirit above or below this standard, as the case may be; and when a numeral is prefixed, it means the number of volumes that are to be added to or subtracted from 100 volumes of the liquid, to bring it to standard or proof strength; thus, 20 over proof means that 100 volumes of liquor require the addition of 20 of water to bring them to proof strength, and when a liquid is 20 under proof, it is understood that 20 parts of water are to be abstracted.

Sikes' hydrometer, a description of which follows, is constructed on this principle. This is selected for special illustration on account of its being the one almost exclusively employed by the revenue in this country.

It consists of a flat stem, A B—Fig. 50—3·4 inches long, which is divided on both sides into 10 or sometimes 11 equal parts, and each of these subdivided into 5, the scale being numbered from zero—0—to 11. The stem, A B, is soldered to a brass ball, 1·6 inch in diameter, into which is fixed a small conical stem, C D, 1·13 inch long; at the end of this is a pear-shaped loaded bulb, D, half an inch in diameter. The whole instrument is made of brass, and is 6·7 inches long. Nine circular weights accompany it, numbered 10, 20, 30, 40, &c., up to 90, and there is another weight in the form of a parallelopiped. Each of the circular weights is cut into the centre, so that they can be placed on the conical stem at C, and slid down to D; in consequence of the enlargement of the cone, they cannot slip off at D, but must be brought up to C for that purpose. The weight in the form of a parallelopiped has a square notch in one of its sides, by which it can be placed on the summit, A, of the stem. In using this instrument, it is immersed in the spirit, and pressed with the finger till sunk to zero, or 0, so as to wet the whole of the stem; from the resistance felt, experience teaches which weight will be required to append to it. After slipping on the weight at C, the instrument is again immersed into the liquid, and pressed with the hand till it has descended to 0 on the scale; the pressure of the hand is then withdrawn, and the apparatus is allowed to emerge and settle at the proper point of the density of the liquid, as indicated by the scale and weights. The figure on the scale to which the hydrometer sinks is now carefully observed, and the weight placed upon the conical stem added thereto; this sum will, by reference to the tables which accompany the instrument, where the same number is found under the column *indication*, and to the temperature which corresponds with that of the liquid under examination, give the percentage of alcohol. The strength is expressed in numbers, denoting the excess or deficiency per cent. of spirit in any sample. Three sliding rules, which are used instead of the tables, likewise accompany the hydrometer. The exact temperature of the liquid should be taken previous to ascertaining the gravity, as the difference in temperature, if not corrected, would give, as the result, a weaker or stronger liquor than if the thermometer stood at 60° Fahr.

Fig. 50.



When excise officers use this instrument they are instructed to take the degree above the mercury, when it stands between any two degrees of the thermometer, as also the division below the surface of the liquid, when it stands between any two lines of the hydrometer, thus giving any advantage the difference occasions to the trader or manufacturer. The square weight or cap shows the difference between the weight of proof spirit and that of water, as described in the first clause of the hydrometer act, being one-twelfth part of the weight of the hydrometer when loaded with the weight 60. If this weight be placed on the top of the stem at A, and the hydrometer be loaded with the weight 60, it will sink in distilled water, at the temperature of 51° Fahr., to the proof point, P, marked on the narrow edge of the stem.

The following will serve as examples to show how the strength of the spirituous liquid is ascertained :—

Example 1.—Suppose the temperature of the solution to be 47°, and that the weight, 60, is required to sink the stem of the hydrometer to 8, which, when added to the weight 60, will give 68 ; then, under the temperature 47° Fahr., and in a line with the marginal indication, section 60, 68 is sought for, and carrying the eye along it is seen that the liquor is 10½ under proof.

Example 2.—If the stem of the instrument, when loaded with 50, sinks to 5, and the temperature, as shown by the thermometer, is 45° Fahr., then in the marginal section headed 50, 55—equal to the weight 50 and the indication on the divided stem—is found; and under the temperature 45°, the noted strength is 10·6 per cent. over proof.

The following are two out of fifty pages in a book given with the instrument :—

40° to 50° Temperature. Sikes' Alcoholometer I. Weight 50.

Indication.	Temperature of the spirits by Fahrenheit's thermometer.											Indication.
	40°	41°	42°	43°	44°	45°	46°	47°	48°	49°	50°	
50	19·2	18·9	18·6	18·3	18·0	17·7	17·3	17·0	16·7	16·4	16·1	50
·2	18·9	18·6	18·3	18·0	17·7	17·4	17·0	16·7	16·4	16·1	15·8	·2
·4	18·7	18·3	18·0	17·7	17·4	17·1	16·8	16·4	16·1	15·8	15·5	·4
·6	18·4	18·1	17·8	17·5	17·2	16·9	16·5	16·2	15·9	15·6	15·3	·6
·8	18·2	17·8	17·5	17·2	16·9	16·6	16·3	15·9	15·6	15·3	15·0	·8
51	17·9	17·5	17·2	16·9	16·6	16·3	16·0	15·6	15·3	15·0	14·7	51
·2	17 6	17·2	16·9	16·6	16·3	16·0	15·7	15·3	15·0	14·7	14·4	·2
·4	17·3	16·9	16·6	16·3	16·0	15·7	15·4	15·1	14·7	14·4	14·1	·4
·6	17·1	16·7	16·4	16·1	15·8	15·5	15·2	14·8	14·5	14·2	13·9	·6
·8	16·8	16·4	16·1	15 8	15·5	15·2	14·9	14·6	14·2	13·9	13 6	·8
52	16·5	16·1	15·8	15·5	15·2	14·9	14·6	14·3	13·9	13·6	13·3	52
·2	16·2	15·8	15·5	15·2	14·9	14·6	14·3	14·0	13·6	13·3	13·0	·2
·4	15·9	15·5	15·2	14·9	14·6	14·3	14·0	13·7	13·3	13·0	12·7	·4
·6	15·6	15·3	15·0	14·7	14·4	14·1	13·7	13·4	13·1	12·8	12·5	·6
·8	15·3	15·0	14·7	14·4	14·1	13·8	13·4	13·1	12·8	12·5	12·2	·8
53	15·0	14·7	14·4	14·1	13·8	13·5	13·1	12·8	12·5	12·2	11·9	53
·2	14·7	14·4	14·1	13·8	13·5	13·2	12·8	12·5	12·2	11·9	11·6	·2
·4	14·4	14·1	13·8	13·5	13·2	12·9	12·5	12·2	11·9	11·6	11·3	·4
·6	14·2	13·9	13·6	13·3	13·0	12·7	12·3	12·0	11·7	11·4	11 1	·6
·8	13·9	13·6	13.3	13·0	12·7	12·4	12·0	11·7	11·4	11·1	10·8	·8
54	13·6	13·3	13·0	12·7	12·4	12·1	11·7	11·4	11·1	10·8	10·5	54
·2	13·3	13·0	12·7	12·4	12·1	11·8	11·4	11·1	10·8	10·5	10·2	·2
·4	13·0	12·7	12·4	12·1	11·8	11·5	11·1	10·8	10·5	10·2	9·9	·4
·6	12·8	12·5	12·2	11·8	11·5	11·2	10·9	10·6	10·2	9·9	9·6	·6
·8	12·5	12·2	11·9	11·5	11·2	10·9	10·6	10·3	9·9	9·6	9·3	·8
55	12·2	11·9	11·6	11·2	10·9	10·6	10·3	10·0	9·6	9·3	9·0	55
·2	11·9	11·6	11·3	10·9	10·6	10·3	10·0	9·7	9·3	9·0	8·7	·2
·4	11·6	11·3	11·0	10·6	10·3	10·0	9·7	9·4	9·0	8·7	8·4	·4
·6	11·3	11·0	10·7	10·4	10·1	9·7	9·4	9·1	8·8	8·5	8·1	·6
·8	11·0	10·7	10·4	10·1	9·8	9·4	9·1	8·8	8·5	8·2	7·8	·8
56	10·7	10·4	10·1	9·8	9·5	9·1	8·8	8·5	8·2	7·9	7·5	56
·2	10·4	10·1	9·8	9·5	9·2	8·8	8·5	8·2	7·9	7·6	7·2	·2
·4	10·1	9·8	9·5	9·2	8·9	8·5	8·2	7·9	7·6	7·3	6·9	·4
·6	9·9	9·6	9·3	9·0	8·6	8·3	7·9	7·6	7·3	7·0	6·7	·6
·8	9·6	9·3	9·0	8·7	8·3	8·0	7·6	7·3	7·0	6·7	6·4	·8
57	9·3	9·0	8·7	8·4	8·0	7·7	7·3	7·0	6·7	6·4	6·1	57
·2	9·0	8·7	8·4	8·1	7·7	7·4	7·0	6·7	6·4	6·1	5·8	·2
·4	8·7	8·4	8·1	7·8	7·4	7·1	6·7	6·4	6·1	5·8	5·5	·4
·6	8·4	8·1	7·8	7·5	7·2	6·8	6·5	6·1	5·8	5·5	5·2	·6
·8	8·1	7·8	7·5	7.2	6·9	6·5	6·2	5·8	5·5	5·2	4·9	·8
58	7·8	7·5	7·2	6·9	6·6	6·2	5·9	5·5	5·2	4·9	4·6	58
·2	7·5	7·2	6·9	6·6	6·3	5·9	5·6	5·2	4·9	4·6	4·3	·2
·4	7·2	6·9	6·6	6·3	6·0	5·6	5·3	4·9	4·6	4·3	4·0	·4
·6	6·9	6·6	6·3	5·9	5·7	5·3	5·0	4·7	4·3	4·0	3·7	·6
·3	6·6	6·3	6·0	5·6	5·4	5·0	4·7	4·4	4·0	3·7	3·4	·8
59	6·3	6·0	5·7	5·3	5·1	4·8	4·4	4·1	3·7	3·4	3·1	59
·2	6·0	5·7	5·4	5·0	4·8	4·4	4·1	3·8	3·4	3·1	2·8	·2
·4	5·7	5·4	5·1	4·7	4·5	4·1	3·8	3·5	3·1	2·8	2·5	·4
·6	5·4	5·1	4·8	4·4	4·1	3·8	3·5	3·2	2·8	2·5	2·1	·6
·8	5·1	4·8	4·5	4·1	3·8	3·5	3·2	2·9	2·5	2·2	1·8	·8
60	4·8	4·5	4·2	3·8	3·5	3·2	2·9	2·6	2·2	1·9	1·5	60
Indication.	40°	41°	42°	43°	44°	45°	46°	47°	48°	49°	50°	Indication.
	Temperature of the spirits by Fahrenheit's thermometer.											

WEIGHT 60. SIKES' ALCOHOLOMETER II. TEMPERATURE 40° TO 50°.

Indication.	Temperature of the spirit by Fahrenheit's thermometer.											Indication.
	40°	41°	42°	43°	44°	45°	46°	47°	48°	49°	50°	
60	4·8	4·5	4·2	3·8	3·5	3·2	2·9	2·6	2·2	1·9	1·5	60
·2	4·5	4·2	3·9	3·5	3·2	2·9	2·6	2·3	1·9	1·6	1·2	·2
·4	4·2	3·9	3·6	3·2	2·9	2·6	2·3	2·0	1·6	1·3	·9	·4
·6	3·9	3·6	3·3	2·9	2·6	2·3	2·0	1·7	1·3	1·0	·6	·6
·8	3·6	3·3	3·0	2·6	2·3	2·0	1·7	1·4	1·0	·7	·3	·8
61	3·3	3·0	2·7	2·3	2·0	1·7	1·4	1·1	·7	·4	·0	61
·2	3·0	2·7	2·4	2·0	1·7	1·4	1·1	·8	·4	·1	·3	·2
·4	2·7	2·4	2·1	1·7	1·4	1·1	·8	·5	·1	·2	·6	·4
·6	2·4	2·1	1·8	1·4	1·1	·8	·4	·1	·3	·6	·9	·6
·8	2·1	1·8	1·5	1·1	·8	·5	·1	·2	·6	·9	1·2	·8
62	1·8	1·5	1·2	·8	·5	·2	·2	·5	·9	1·2	1·5	62
·2	1·5	1·2	·9	·5	·2	·1	·5	·8	1·2	1·5	1·8	·2
·4	1·2	·9	·6	·2	·1	·4	·8	1·1	1·5	1·8	2·1	·4
·6	·8	·5	·2	·2	·5	·8	1·1	1·5	1·8	2·2	2·5	·6
·8	·5	·2	·1	·5	·8	1·1	1·4	1·8	2·1	2·5	2·8	·8
63	·2	·1	·4	·8	1·1	1·4	1·7	2·1	2·4	2·8	3·1	63
·2	·1	·4	·7	1·1	1·4	1·7	2·0	2·4	2·7	3·1	3·4	·2
·4	·4	·7	1·0	1·4	1·7	2·1	2·4	2·7	3·0	3·4	3·7	·4
·6	·8	1·1	1·4	1·8	2·1	2·4	2·7	3·1	3·4	3·8	4·1	·6
·8	1·1	1·4	1·7	2·1	2·4	2·8	3·1	3·4	3·7	4·1	4·1	·8
64	1·4	1·7	2·0	2·4	2·7	3·1	3·4	3·7	4·0	4·4	4·1	64
·2	1·7	2·0	2·3	2·7	3·0	3·4	3·7	4·0	4·3	4·7	5·6	·2
·4	2·0	2·3	2·6	3·0	3·3	3·7	4·1	4·4	4·7	5·1	5·1	·4
·6	2·4	2·7	3·0	3·4	3·7	4·1	4·4	4·7	5·0	5·4	5·7	·6
·8	2·7	3·0	3·3	3·7	4·0	4·4	4·8	5·1	5·4	5·8	6·1	·8
65	3·0	3·3	3·6	4·0	4·3	4·7	5·1	5·4	5·7	6·1	6·4	65
·2	3·3	3·6	3·9	4·3	4·6	5·0	5·4	5·7	6·0	6·4	6·7	·2
·4	3·6	4·0	4·3	4·7	5·0	5·4	5·7	6·1	6·4	6·8	7·1	·4
·6	4·0	4·3	4·6	5·0	5·3	5·7	6·1	6·4	6·7	7·1	7·4	·6
·8	4·3	4·7	5·0	5·4	5·7	6·1	6·4	6·8	7·1	7·5	7·8	·8
66	4·6	5·0	5·3	5·7	6·0	6·4	6·7	7·1	7·4	7·8	8·1	66
·2	5·0	5·3	5·7	6·0	6·3	6·7	7·0	7·4	7·7	8·1	8·4	·2
·4	5·3	5·7	6·0	6·4	6·7	7·1	7·4	7·8	8·1	8·5	8·8	·4
·6	5·7	6·0	6·4	6·7	7·0	7·4	7·7	8·1	8·4	8·8	9·1	·6
·8	6·0	6·4	6·7	7·1	7·4	7·8	8·1	8·5	8·8	9·2	9·5	·8
67	6·4	6·7	7·1	7·4	7·7	8·1	8·4	8·8	9·1	9·5	9·8	67
·2	6·7	7·0	7·4	7·7	8·0	8·4	8·7	9·1	9·4	9·8	10·1	·2
·4	7·1	7·4	7·8	8·1	8·4	8·8	9·1	9·5	9·8	10·2	10·5	·4
·6	7·4	7·7	8·1	8·4	8·7	9·1	9·4	9·8	10·1	10·5	10·8	·6
·8	7·8	8·1	8·5	8·8	9·1	9·5	9·8	10·2	10·5	10·9	11·2	·8
68	8·1	8·4	8·8	9·1	9·4	9·8	10·1	10·5	10·8	11·2	11·5	68
·2	8·5	8·8	9·1	9·4	9·8	10·2	10·5	10·9	11·2	11·6	11·9	·2
·4	8·8	9·1	9·5	9·8	10·1	10·5	10·8	11·2	11·5	11·9	12·2	·4
·6	9·2	9·5	9·8	10·1	10·5	10·9	11·2	11·6	11·9	12·3	12·6	·6
·8	9·5	9·8	10·2	10·5	10·8	11·2	11·5	11·9	12·2	12·6	12·9	·8
69	9·9	10·2	10·5	10·8	11·2	11·6	11·9	12·3	12·6	13·0	13·3	69
·2	10·3	10·6	10·9	11·2	11·6	12·0	12·3	12·7	13·0	13·4	13·7	·2
·4	10·6	10·9	11·2	11·5	11·9	12·3	12·6	13·0	13·3	13·7	14·0	·4
·6	11·0	11·3	11·6	11·9	12·3	12·7	13·0	13·4	13·7	14·1	14·4	·6
·8	11·3	11·6	11·9	12·2	12·6	13·0	13·3	13·7	14·0	14·4	14.7	·8
70	11·7	12·0	12·3	12·6	13·0	13·4	13·7	14·1	14·4	14·8	15·1	70
Indication.	40°	41°	42°	43°	44°	45°	46°	47°	48°	49°	50°	Indication.
	Temperature of the spirit by Fahrenheit's thermometer.											

A modification of Sikes' hydrometer has been constructed of glass, and is in the shape of an ordinary hydrometer, the stem being divided into degrees from 1 to 100; it carries a small delicate spirit thermometer in the bulb, to which a scale is affixed, having references from 0° to 12°, corresponding to Fahrenheit's scale from 32° to 80°. The temperature of the alcoholic liquors is indicated by the small thermometer; and the calculations in the tables accompanying the instrument are made to accord with these numbers. Tables are supplied with the hydrometer, which are headed by the degrees and half degrees of the thermometric scale; and the corresponding amount of spirit over or under proof at the respective degree of the table is placed opposite each degree of the hydrometer.

When the instrument is used to ascertain the value of a liquid, the degree of immersion and also that of the thermometer are noted; on referring to the table headed by the observed temperature, the percentage of spirit over or under proof is found opposite the degree to which the hydrometer sunk in the liquid:

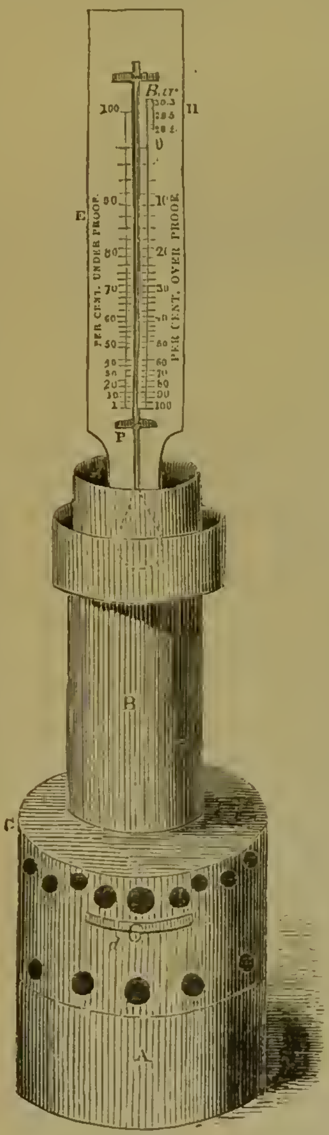
By Sikes' hydrometer, the specific gravity of the spirit indicating proof strength is ·9200. Ure has constructed a table, appended below, wherein the specific gravity corresponds to the strength noted by the hydrometer, whether over or under proof strength.

CORRESPONDENCE BETWEEN THE SPECIFIC GRAVITIES AND PER CENTS. OF ALCOHOL OVER PROOF AT 60° FAHRENHEIT.

Specific gravity.	Per cent. over proof.	Specific gravity.	Per cent. over proof.	Specific gravity.	Per cent. over proof.	Specific gravity.	Per cent. over proof.	Specific gravity.	Per cent. over proof.	Specific gravity.	Per cent. under proof.	Specific gravity.	Per cent. under proof.
0·8156	67·0	0·8410	54·2	0·8657	39·3	0·8912	21·9	0·9178	1·9	0·9445	21·9	0·9722	58·3
8160	66·8	8413	54·1	8660	39·1	8915	21·7	9182	1·6	9448	22·2	9726	59·0
8163	66·6	8417	53·9	8664	38·9	8919	21·4	9185	1·3	9452	22·7	9730	59·7
8167	66·5	8420	53·7	8667	38·7	8922	21·2	9189	1·0	9456	23·1	9734	60·4
8170	66·3	8424	53·5	8671	38·4	8926	20·9	9192	0·7	9460	23·5	9738	61·1
8174	66·1	8427	53·3	8674	38·2	8930	20·6	9196	0·3	9464	23·9	9742	61·8
8178	65·6	8431	53·1	8678	38·0	8933	20·4	9200	Proof.	9468	24·3	9746	62·5
8181	65·8	8434	52·9	8681	37·8	8937	20·1	Under proof.		9472	24·7	9750	63·2
8185	65·6	8438	52·7	8685	37·6	8940	19·9	9204	0·3	9476	25·1	9754	63·9
8188	65·5	8441	52·5	8688	37·3	8944	19·6	9207	0·6	9480	25·5	9758	64·6
8192	65·3	8445	52·3	8692	37·1	8948	19·3	9210	0·9	9484	25·9	9762	65·3
8196	65·1	8448	52·1	8695	36·9	8951	19·1	9214	1·3	9488	26·3	9766	66·0
8199	65·0	8452	51·9	8699	36·7	8955	18·8	9218	1·6	9492	26·7	9770	66·7
8203	64·8	8455	51·7	8702	36·4	8959	18·6	9222	1·9	9496	27·1	9774	67·4
8206	64·7	8459	51·5	8706	36·2	8962	18·3	9226	2·2	9499	27·5	9778	68·0
8210	64·5	8462	51·3	8709	35·9	8966	18·0	9229	2·5	9503	28·0	9782	68·7
8214	64·3	8465	51·1	8713	35·7	8970	17·7	9233	2·8	9507	28·4	9786	69·4
8218	64·1	8469	50·9	8716	35·5	8974	17·5	9237	3·1	9511	28·8	9790	70·1
8221	64·0	8472	50·7	8720	35·2	8977	17·2	9241	3·4	9515	29·2	9794	70·8
8224	63·8	8476	50·5	8723	35·0	8981	16·9	9244	3·7	9519	29·7	9798	71·4
8227	63·6	8480	50·3	8727	34·7	8985	16·6	9248	4·0	9522	30·1	9802	72·1
8231	63·4	8482	50·1	8730	34·5	8989	16·4	9252	4·4	9526	30·6	9806	72·8
8234	63·2	8486	49·9	8734	34·3	8992	16·1	9255	4·7	9530	31·0	9810	73·5
8238	63·1	8490	49·7	8737	34·1	8996	15·9	9259	5·0	9534	31·4	9814	74·1
8242	62.9	8493	49·5	8741	33·8	9000	15·6	9263	5·3	9539	31·1	9818	74·8
8245	62·7	8496	49·3	8744	33·6	9004	15·3	9267	5·7	9542	32·3	9822	75·4
8249	62·5	8499	49·1	8748	33·4	9008	15·0	9270	6·0	9546	32·8	9826	76·1
8252	62·3	8503	48·9	8751	33·2	9011	14·8	9274	6·4	9550	33·2	9830	76·7
8256	62·2	8506	48·7	8755	32·9	9015	14·5	9278	6·7	9553	33·7	9834	77·3
8259	62·0	8510	48·5	8758	32·7	9019	14·2	9282	7·0	9557	34·2	9838	78·0
8263	61·8	8513	48.3	8762	32·4	9023	13·9	9286	7·3	9561	34·7	9842	78·6
8266	61·6	8516	48·0	8765	32·2	9026	13·6	9291	7·7	9565	35·1	9846	79·2
8270	61·4	8520	47·8	8769	32·0	9030	13·4	9295	8·0	9569	35·6	9850	79·8
8273	61·3	8523	47·6	8772	31·7	9034	13·1	9299	8·3	9573	36·1	9854	80·4
8277	61·1	8527	47·4	8776	31·5	9038	12·8	9302	8·6	9577	36·6	9858	81·1
8280	60·9	8530	47·2	8779	31·2	9041	12·5	9306	9·0	9580	37·1	9862	81·7
8284	60·7	8533	47·0	8783	31·0	9045	12·2	9310	9·3	9584	37·6	9866	82·3
8287	60·5	8537	46·8	8786	30·8	9049	12·0	9314	9·7	9588	38·1	9870	82·9
8291	60·4	8540	46·6	8790	30·5	9052	11·7	9318	10·0	9592	38·6	9874	83·5
8294	60·2	8543	46·4	8793	30·3	9056	11·4	9322	10·3	9596	39·1	9878	84·0
8298	60·0	8547	46·2	8797	30·0	9060	11·1	9326	10·7	9599	39·6	9882	84·6
8301	59·8	8550	46·0	8800	29·8	9064	10·8	9329	11·0	9603	40·1	9886	85·2
8305	59·6	8553	45·8	8804	29·5	9067	10·6	9332	11·4	9607	40·6	9890	85·8
8308	59·5	8556	45·6	8807	29·3	9071	10·3	9337	11·7	9611	41·1	9894	86·3
8312	59·3	8560	45·4	8811	29·0	9075	10·0	9341	12·1	9615	41·7	9898	86·9
8315	59·1	8563	45·2	8814	28·8	9079	9·7	9345	12·4	9619	42·2	9902	87·4
8319	58·9	8566	45·0	8818	28·5	9082	9·4	9349	12·8	9623	42·8	9906	88·0
8322	58·7	8570	44·8	8822	28·3	9085	9·2	9353	13·1	9627	43·3	9910	88·5
8326	58·6	8573	44·6	8825	28·0	9089	8·9	9357	13·5	9631	43·9	9914	89·1
8329	58·4	8577	44·4	8829	27·8	9093	8·6	9360	13·9	9635	44·4	9918	89·6
8333	58·2	8581	44·2	8832	27·5	9097	8·3	9364	14·2	9638	45·0	9922	90·2
8336	58·0	8583	43·9	8836	27·3	9100	8·0	9368	14·6	9642	45·5	9926	90·7
8340	57·8	8587	43·7	8840	27·0	9104	7·7	9372	14·9	9646	46·1	9930	91·2
8344	57·7	8590	43·5	8843	26·8	9107	7·4	9376	15·3	9650	46·7	9934	91·7
8347	57·5	8594	43·3	8847	26·5	9111	7·1	9380	15·7	9654	47·3	9938	92·3
8351	57·3	8597	43·1	8850	26·3	9115	6·8	9384	16·0	9657	47·9	9942	92·8
8354	57·1	8601	42·8	8854	26·0	9118	6·5	9388	16·4	9661	48·5	9946	93·3
8358	56·9	8604	42·6	8858	25·8	9122	6·2	9392	16·7	9665	49·1	9950	93·8
8362	56·8	8608	42·4	8861	25·5	9126	5·9	9396	17·1	9669	49·7	9954	94·3
8365	56·6	8611	42·2	8865	25·3	9130	5·6	9399	17·5	9674	50·3	9958	94·9
8369	56·4	8615	42·0	8869	25·0	9134	5·3	9403	17·8	9677	51·0	9962	95·4
8372	56·2	8618	41·7	8872	24·8	9137	5·0	9407	18·2	9681	51·6	9966	95·9
8376	56·0	8622	41·5	8876	24·5	9141	4·8	9411	18·5	9685	52·2	9970	96·4
8379	55·9	8625	41·3	8879	24·3	9145	4·5	9415	18·9	9689	52·9	9974	96·8
8383	55·7	8629	41·1	8883	24·0	9148	4·2	9419	19·3	9693	53·3	9978	97·3
8386	55·5	8632	40·9	8886	23·8	9152	3·9	9422	19·7	9697	54·2	9982	97·7
8390	55·3	8636	40·6	8890	23·5	9156	3·6	9426	20·0	9701	54·8	9986	98·2
8393	55·1	8639	40·4	8894	23·2	9159	3·3	9430	20·4	9705	55·5	9990	98·7
8396	55·0	8643	40·2	8897	23·0	9163	3·0	9434	20·8	9709	56·2	9993	99·1
8400	54·8	8646	40·0	8901	22·7	9167	2·7	9437	21·2	9713	56·9	9997	99·6
8403	54·6	8650	39·8	8904	22·5	9170	2·4	9441	21·6	9718	57·6	1·0000	100·0
8407	54·4	8653	39·6	8908	22·2	9174	2·1						

Although it had long been known that the boiling point of water was raised by the solution in it of neutral salts and other bodies, the application of this fact to the determination of the quantity of alcohol in spirituous liquors was reserved for the Abbé Brossard-Vidal, of Toulon, who ascertained that the boiling point of such liquors was in direct proportion to the quantity of alcohol they contained, irrespective of any amount of saline ingredients present in them. When such salts are added to dilute alcohol as abstract a portion of the water, the boiling point of the solution is lowered, instead of the reverse being the case.

Fig. 51.



This principle also formed the basis of Field's alcoholometer, for the determination of the amount of spirit in every description of alcoholic liquor at any period after fermentation, for which he obtained a patent in 1847. It consisted originally of a spirit lamp, surmounted by a boiler containing the liquid to be examined, and a large cylindrical glass bulb containing mercury, and having an upright stem, whose calibre was such as to allow the expansion of the mercury to elevate a small glass float which rested on its surface. This float was connected by a thread with a similar bead, which hung in the open air; the thread passed over a pulley which, turning with the motion of the beads, caused the index to move along the circular graduated scale. This instrument was much improved by Ure, who introduced, instead of the cylindrical bulb and beads, a thermometer attached to a graduated scale. Fig. 51 shows the improved instrument.

A is the spirit lamp, surrounded by an outer coating containing cold water for keeping the lamp cool, should many successive experiments be required; B is the boiler, which fits into the cage, C, in the case of the lamp; a damper, *d*, regulates the heat of the lamp; E is the thermometer, which has a very small bore. The bottom of the scale on the ebullition thermometer is marked P, for proof, on the left side, and 100—proof spirit—on the right side. It corresponds with 178·6 Fahr. nearly, or the boiling point of alcohol, spec. grav. ·920.

From the mean of a great number of experiments, Ure drew up the following table, which shows the boiling point of alcohol of various specific gravities:—

Temperature Fahr.	Specific gravity.		
178·5	0·9200	—	Proof.
179·75	0·9321	10	Under proof.
180·4	0·9420	20	"
182·01	0·9516	30	"
183·40	0·960	40	"
185·6	0·9665	50	"
189·0	0·9729	60	"
191·8	0·9786	70	"
196·4	0·9850	80	"
202·0	0·992	90	"

When the spirit is above proof, the variation of its boiling point is so small that a strictly accurate result cannot be obtained: proof spirit, or spirit approaching to that strength, is more accurately tested by diluting it with its own bulk of water before ascertaining the strength, and then doubling the result. Another source of error pointed out by Ure, is the elevation of the boiling point when the liquor is kept heated for any length of time; this is, however, obviated by the addition of common salt to the solution in the boiler of the apparatus. In order to correct the difference arising from a higher or lower pressure of the atmosphere, distilled water should be boiled in the apparatus, and the temperature noted; for the boiling point of the alcoholic solutions will vary as that of the water, when the pressure of the atmosphere is greater or less. In order to correct this source of error, a subsidiary barometrical scale, H, is attached to the thermometer, F. The method of using this alcoholometer is as follows:—

The lamp, A, is lighted; B is filled to about one inch from the top with the liquid to be examined, to which a paper of common salt is added, and the whole is then placed on the lamp, A; and lastly, the thermometer, E, is fixed to the scale, with its bulb immersed in the liquid. Before commencing general operations for the day, the boiler, B, is filled with water, and boiled; if the column of mercury remains at 29·5, which is placed opposite to 100 on the scale at the left hand—the mean boiling point of water—no correction is required to be made; but if it stands higher or lower than this figure, the various boiling points of the samples bear reference to this. In testing the solutions, when the mercury begins to rise out of the bulb of the thermometer, the damper is to be pushed in midway into the groove to lower the heat. As the liquid boils freely, the mercury in the tube will become stationary, and the figure on the left will indicate the percentage under proof, while that on the right will show the percentage over proof. Field's alcoholometer only tells the quantity of alcohol, but an auxiliary experiment with the hydrometer will readily give the specific gravity, and upon reference to Table I., the amount of saccharine and extractive matter can be ascertained. In testing wines, the specific gravity of the liquid is first taken, then the boiling point ascertained by the alcoholometer, and the corresponding specific gravity deducted from that previously found by the hydrometer; the difference will, upon referring to Table II., show the quantity of extractive and saccharine matter in 100 gallons of the liquid. For example, if the specific gravity by the hydrometer be ·989, and that by the alcoholometer shows the presence of alcohol of 69$\frac{6}{10}$, whose specific gravity is ·979 by the tables, deduct the latter gravity from the gravity of the bulk, or ·989, and a difference of ten remains, which number, upon reference to Table II., will give 25 lbs. of saccharine and extractive matter in 100 gallons, combined with 30$\frac{4}{10}$ gallons of proof spirit. Should the barometer be above or below the standard 29·5, the thermometer scale will then only show the apparent strength, and reference must be had to the small ivory indicator, it being the counterpart of the barometrical scale of the thermometer; thus, if the barometer indicate 30, place 30 of the indicator against the boiling point of the liquid, and opposite the line of 29·5 will be found the true strength.

Example 1.—Barometer at 30.—Suppose the mercury to stop at the same point, 72 U.P.; place 30 of the indicator against 72 on the thermometer, and the line 29·5 will cut 69·6 U.P., the true percentage.

Example 2.—Barometer at 29.—Suppose the mercury to stop at the same point, 72 U.P.; place 29 of the indicator against 72 on the thermometer, and the line 29·5 will cut 74·3 U.P., the true strength.

To ascertain the strength of malt liquors and their respective values, the instrument has been furnished with a glass saccharometer, testing glass, and slide rule. Commence by charging the test glass with the liquid, then insert the saccharometer to ascertain the present gravity or density per barrel, the number at which it floats indicates the number of pounds per barrel the liquor is heavier than water:—

Example 1.—Suppose the saccharometer to float at the figure 8, which would indicate 8 lbs. per barrel; then submit the liquid to the boiling test, with an addition of salt to arrest the mercury at the true boiling point of the liquid; now, suppose it should show—the barometrical difference being accounted for—90 U.P., that would be equivalent to 10 per cent. of proof alcohol. Refer to the slide scale, and place A on the slide against 10 on the upper line of figures, and facing B on the lower line will be 18, showing that 18 lbs. per barrel have been decomposed to constitute that percentage of spirit; then, by adding 18 lbs. to the present 8 lbs. per barrel, the result will be 26 lbs., the original weight of the wort after leaving the copper.

Example 2.—The saccharometer marks 10 lbs. per barrel, and at the boiling point it indicates 88 U.P., equivalent to 12 gallons of proof spirit per cent.; place A against 12, and opposite B will be 21½, the number of lbs. per barrel, which, when added to the 10 lbs. present, will give a total of 31½ lbs.

To ascertain the relative value.—Suppose the price of the 26 lbs. beer to be 36*s.* per barrel, and the 31½ lbs. beer to be 40*s.*—to ascertain which beer will be the cheaper, place 26 on the opposite side of the rule against 36, and opposite 31½ lbs. will be 43*s.* 7*d.*, showing that the latter beer is cheaper by 3*s.* 7*d.* per barrel. By this instrument the quantity of spirit per cent. in distiller's wort, whether it be in progress of fermentation or ready for the still, is indicated, the only difference being in the allowances in the sliding rule. Saccharometers applicable to the foregoing rules for beer, ale, &c., have been adjusted at the temperature of 60° Fahr., and will be found sufficiently correct for general purposes; but where extreme minuteness is required, the variation of temperature must be taken into account, and for every 10 degrees of temperature above 60°, three-tenths of a pound must be added to the gross amount found by the slide rule; on the other hand, for every 10 degrees below 60°, three-tenths of a pound must be deducted.

For cordialized spirits the operation is different from that applied to the testing of beers, which have the alcohol generated in the wort. In cordials of every kind, the alcohol is the original, and the saccharine matter, or sugar, is an addendum. If 100 gallons of spirit are required of a given strength—say 50 per cent. under proof—50 gallons of proof spirit to which 50 gallons of water are added will be of this strength, and upon testing it by the alcoholometer it will be found as correct as by the hydrometer. But in cordializing spirit, the reverse is the case; for to the 50 gallons of spirit, proof strength, 50 gallons of sugar and water would be added, thereby rendering the hydrometer useless, excepting for taking the specific gravity of the bulk, and, according to the quantity of sugar present, so a relative quantity of water must have been displaced; and as the sugar has no reducing properties, the alcoholometer will only show the strength of the cordial in relation to the quantity of water contained in it, as the principle indicates, irrespective of the saccharine or extractive matter. Suppose, in making 100 gallons of cordial at 50 U.P., 3 lbs. of sugar are put to the gallon, or 300 lbs. to the 100 gallons, these 300 lbs. displacing 18·67 gallons of water, only 31·33 gallons of the water, instead of 50, have been applied; the sugar, without reducing properties, making up the bulk of 100 gallons, which is meant to represent 50 per cent. U.P.

The alcoholometer will only show at the full point of ebullition the alcoholic strength in relation to the water in 100 gallons of the mixture, or 35 per cent. U.P., leaving 15 per cent. to be accounted for in the bulk. As the quantity of sugar present must be determined before that percentage can be arrived at, a double object will be effected by so doing, namely, eliciting in all instances the quantity of sugar present, as well as the percentage of spirit to be accounted for.

Example.—Suppose the specific gravity of a cordial is 1·076, then submit the liquid to the boiling point, and having ascertained the percentage of alcohol—35 U.P.—the specific gravity of alcohol at that strength will be found to be 0·956; deduct 0·956 from the specific gravity of the bulk, or 1·076, and ·120 will remain; refer that to its amount in the head line of Table II., namely, 120, under which will be found 3, representing 3 lbs. of sugar to the gallon; and by running the eye down its column to opposite the alcoholic strength indicated—35 U.P.—will be found 14·9, representing the water displaced by the sugar, and which amount added to 35 per cent. ascertained, makes the total upon the bulk 49·9 per cent. U.P., with 3 lbs. of sugar to the gallon.

Table I., which shows the specific gravity on the bulk of the mixture, bears reference to the table following it—Table II. of the alcoholometer.

In estimating the density of various gins and cordials, suppose the specific gravity of the liquid is found to be ·957, and by the boiling point it proves to be 14 U.P., whose specific gravity is ·937; when this is deducted from the former—·957—the remainder is 20, under which in Table II. will be found ½, or one-half pound of sugar to the gallon; and on observing the opposite 14 U.P., 3·0 will be found, which, added to 14, makes the total on the bulk 17 per cent. U.P., with 50 pounds of sugar to the 100 gallons.

TABLE I.

Table of specific gravities by Sikes' hydrometer, adapted to Field's alcoholometer for cordialized spirits.

Temperature, 60°.—Specific gravity of water, 1·000°.

60		70		80		90		100		110		120		130		140		150		160		170		180	
Wt.	S. G.	Wt.	S. G.	Wt.	S. G.	Wt.	S. G.	Wt.	S. G.	Wt.	S. G.	Wt.	S. G.	Wt.	S. G.	Wt.	S. G.	Wt.	S. G.	Wt.	S. G.	Wt.	S. G.	Wt.	S. C.
60	922	70	942	80	961	90	981	100	1000	110	1020	120	1041	130	1063	140	1085	150	1107	160	1129	170	1152	180	1175
1	924	1	943	1	963	1	983	1	1002	1	1022	1	1044	1	1065	1	1087	1	1109	1	1131	1	1155	1	1178
2	926	2	945	2	965	2	985	2	1004	2	1024	2	1046	2	1067	2	1089	2	1111	2	1134	2	1157	2	1180
3	928	3	947	3	967	3	987	3	1006	3	1026	3	1048	3	1069	3	1091	3	1113	3	1136	3	1159	3	1182
4	930	4	949	4	969	4	989	4	1008	4	1029	4	1050	4	1071	4	1093	4	1116	4	1139	4	1162	4	1185
5	932	5	951	5	971	5	991	5	1010	5	1031	5	1052	5	1074	5	1096	5	1118	5	1141	5	1164	5	1187
6	934	6	953	6	973	6	993	6	1012	6	1033	6	1054	6	1076	6	1098	6	1120	6	1143	6	1166	6	1189
7	936	7	955	7	975	7	995	7	1014	7	1035	7	1056	7	1078	7	1100	7	1123	7	1145	7	1168	7	1191
8	938	8	957	8	977	8	997	8	1016	8	1037	8	1058	8	1080	8	1102	8	1125	8	1148	8	1171	8	1194
9	940	9	959	9	979	9	999	9	1018	9	1039	9	1061	9	1082	9	1104	9	1127	9	1150	9	1173	9	1196
70	942	80	961	90	981	100	1000	110	1020	120	1041	130	1063	140	1085	150	1107	160	1129	170	1152	180	1175	190	1199

TABLE II.

Table showing the lbs. of sugar per gallon in cordialized spirits, with the per centages to be added to the indicated strength, per the alcoholometer.

Difference of gravity. Lbs. of sugar per gallon.		10 4 oz. or 25 to 100.	15 6 oz. 37½ to 100.	20 8 oz. 50 to 100.	25 10 oz. 62½ to 100.	30 12 oz. 75 to 100.	35 14 oz. 87½ to 100.	40 1·0	45 oz. 1·2	60 oz. 1·4	Difference of gravity. Lbs. of sugar per gallon.	
Sp. grav. of spirit.	Per cent. of spirit.										Per cent. of spirit.	Sp. grav. of spirit.
920	Proof.	1·6	2·5	3·4	4·4	5·3	6·2	7·1	8·1	9·0	Proof.	920
923	2·5	1·6	2·5	3·3	4·3	5·2	6·1	6·9	7·8	8·8	2·5	923
926	5·	1·5	2·4	3·2	4·2	5·0	5·9	6·8	7·7	8·6	5·	926
929	7·5	1·5	2·3	3·2	4·1	4·9	5·8	6·6	7·5	8·4	7·5	929
932	10·	1·4	2·2	3·1	4·0	4·8	5·7	6·5	7·4	8·2	10·	932
935	12·5	1·4	2·2	3·1	3·9	4·7	5·5	6·3	7·2	8·0	12·5	935
938	15·	1·4	2·1	3·0	3·8	4·6	5·4	6·2	7·0	7·8	15·	938
940	17·5	1·3	2·1	2·9	3·7	4·5	5·3	6·0	6·8	7·6	17·5	940
943	20·	1·3	2·0	2·8	3·6	4·4	5·2	5·9	6·7	7·5	20·	943
945	22·5	1·3	2·0	2·7	3·5	4·3	5·0	5·7	6·5	7·3	22·5	945
948	25·	1·2	1·9	2·6	3·4	4·1	4·8	5·5	6·3	7·0	25·	948
950	27·5	1·2	1·9	2·5	3·3	4·0	4·7	5·3	6·1	6·8	27·5	950
952	30·	1·1	1·8	2·4	3·1	3·8	4·5	5·1	5·8	6·5	30·	952
954	32·5	1·1	1·7	2·3	3·0	3·6	4·3	4·8	5·5	6·2	32·5	954
956	35·	1·0	1·6	2·2	2·9	3·5	4·1	4·6	5·3	6·0	35·	956
958	37·5	1·0	1·6	2·1	2·8	3·4	3·9	4·4	5·1	5·8	37·5	958
960	40·	·9	1·5	2·0	2·7	3·2	3·8	4·3	4·9	5·5	40·	960
962	42·5	·9	1·5	2·0	2·6	3·1	3·6	4·1	4·7	5·3	42·5	962
964	45·	·9	1·4	1·9	2·5	3·0	3·5	4·0	4·6	5·1	45·	964
965	47·5	·8	1·4	1·9	2·4	2·9	3·4	3·9	4·4	4·9	47·5	965
967	50·	·8	1·3	1·8	2·3	2·8	3·3	3·8	4·3	4·8	50·	967
969	52·5	·7	1·2	1·7	2·2	2·6	3·1	3·6	4·1	4·5	52·5	969
970	55·	·7	1·2	1·6	2·0	2·4	2·9	3·4	3·8	4·2	55·	970
972	57·5	·6	1·1	1·5	1·9	2·2	2·7	3·1	3·5	3·9	57·5	972
973	60·	·6	1·0	1·4	1·8	2·1	2·5	2·9	3·3	3·6	60·	973
974	62·5	·6	1·0	1·3	1·7	2·0	2·4	2·7	3·1	3·5	62·5	974
976	65·	·5	·9	1·2	1·5	1·8	2·2	2·5	2·8	3·1	65·	976
977	67·5	·5	·8	1·1	1·4	1·7	2·0	2·3	2·6	2·9	67·5	977
979	70·	·4	·7	1·0	1·3	1·5	1·8	2·1	2·4	2·6	70·	979
980	72·5	·4	·7	·9	1·1	1·3	1·6	1·9	2·1	2·3	72·5	980
982	75·	·3	·6	·8	1·0	1·2	1·4	1·6	1·8	2·0	75·	982
983	77·5	·3	·5	·7	·9	1·0	1·2	1·4	1·6	1·8	77·5	983
984	80·	·2	·4	·6	·8	·9	1·0	1·2	1·4	1·6	80·	984
986	82·5	·2	·3	·5	·7	·8	·9	1·0	1·2	1·4	82·5	986
988	85·	·2	·2	·4	·6	·7	·8	·9	1·0	1·2	85·	988
990	87·5	·1	·2	·3	·5	·6	·7	·8	·9	1·0	87·5	990
992	90·	·1	·1	·2	·4	·5	·6	·7	·8	·9	90·	992
994	92·5	..	·1	·2	·3	·4	·5	·6	·7	·8	92·5	994
996	95·	..	..	·1	·2	·3	·4	·5	·6	·7	95·	996
998	97·5	..	..	..	·1	·2	·3	·4	·5	·6	97·5	998

Care must be taken that the mercury is entirely in the bulb of the thermometer before it is fixed to the stem; and salt must always be added when determining the boiling point, except in the case of water.

The instrument is very useful for testing the relative quantity of fruit and alcohol in normal wines.

FOWNES drew up a table of the value, in absolute alcohol, of spirits of different specific gravities, which is given below.

The table was formed synthetically; absolute alcohol and distilled water were weighed out in the required proportions, mixed in small closely-stopped bottles, and well shaken together. After standing three or four days, the mixtures were brought to the temperature of 60° Fahr. exactly, and their specific gravities determined with great care. When two or three days more had elapsed, the specific gravity was again taken, but in no case had any further contraction occurred. Neither was the specific gravity of a mixture, containing nearly equal parts alcohol and water, which had been so examined, changed by being inclosed in a strong accurately-stoppered bottle, and heated for some time to a temperature above its boiling point.

In this manner, every alternate number in the table —every even number—was obtained by direct experiment; the others were then incorporated.

TABLE OF THE PROPORTION BY WEIGHT

Of real or absolute alcohol contained in 100 parts of spirits of different specific gravities, at the temperature of 60° Fahr.

Specific gravity.	Per centage of alcohol.	Specific gravity.	Per centage of alcohol.	Specific gravity.	Per centage of alcohol.
·9991	0·5	·9511	34	·8769	68
·9981	1	·9490	35	·8745	69
·9965	2	·9470	36	·8721	70
·9947	3	·9452	37	·8696	71
·9930	4	·9434	38	·8672	72
·9914	5	·9416	39	·8649	73
·9898	6	·9396	40	·8625	74
·9884	7	·9376	41	·8603	75
·9869	8	·9356	42	·8581	76
·9855	9	·9335	43	·8557	77
·9841	10	·9314	44	·8533	78
·9828	11	·9292	45	·8508	79
·9815	12	·9270	46	·8183	80
·9802	13	·9249	47	·8459	81
·9789	14	·9228	48	·8434	82
·9778	15	·9206	49	·8408	83
·9766	16	·9184	50	·8382	84
·9753	17	·9160	51	·8357	85
·9741	18	·9135	52	·8331	86
·9728	19	·9113	53	·8305	87
·9716	20	·9090	54	·8279	88
·9701	21	·9069	55	·8254	89
·9691	22	·9047	56	·8228	90
·9678	23	·9025	57	·8199	91
·9665	24	·9001	58	·8172	92
·9652	25	·8979	59	·8145	93
·9638	26	·8956	60	·8118	94
·9623	27	·8932	61	·8089	95
·9609	28	·8908	62	·8061	96
·9593	29	·8886	63	·8031	97
·9578	30	·8863	64	·8001	98
·9560	31	·8840	65	·7969	99
·9544	32	·8816	66	·7938	100
·9528	33	·8793	67		

The contraction of volume suffered by various mixtures of alcohol and water, may be rendered obvious by comparing the actual specific gravities of such mixtures with their calculated mean densities. In Fig. 52, on the next page, in which the vertical lines represent the percentage of alcohol by weight, and the horizontal lines the specific gravities, the calculated mean specific gravities of the mixtures are seen to form a straight diagonal line from corner to corner, while the actual densities form an irregular curve with upward convexity, rising quickly to its maximum deviation at 30 per cent., running almost parallel with the other line to 50 per cent., and thence declining until it reaches the extremity of the scale.

SILBERMANN holds that the various means employed for ascertaining the respective quantities of alcohol and water in mixtures of those liquids possess many disadvantages. The distillation process is rarely employed, on account of the great skill required in its application, and the length of time the operation demands before the results are arrived at. This method, at best, is only applied where accurate and scientific truth is required, irrespective of delay and toil. The density test is open to error on account of extractive matters being present, which render the specific gravity of the impregnated liquid higher than that of alcohol, and thus prevent the true amount of spirit from being determined with strict accuracy; hence the excise duty is, in consequence, evaded. Wines tested by the density process only indicate about one-half their strength; and for this reason GAY-LUSSAC combines, with the use of his alcoholometric areometer, the distillation test.

He objects likewise to the boiling test of Field's hydrometer, that liquids may be heated beyond their boiling point without ebullition, and that the thermometer, even when immersed in the liquid, may under certain circumstances stand several degrees above the real temperature, which would give rise to a difference in result of four alcoholometric degrees for every such extra indication. He also considers that the barometric variations should be taken into account in some better way. SILBERMANN'S arrangement to obviate these evils is based upon the dilatation of the alcoholic liquid. It is well known that between 32° and 212° Fahr. (0° and 100° C.), the dilatation of alcohol is triple that of water. This is much greater between 77° and 122° Fahr. (25° and 50° C.)—and may thus be demonstrated:—Pour water at 25° C. into a thermometer tube, so as to fill the reservoir, and a small portion of the tube up to a certain mark; then, on heating the thermometer to 50° C. the water will rise a certain distance above the mark, and let this point be scratched on the tube; now, if the same quantity of pure alcohol, also at 25° C., be substituted for the water, and heated to the temperature of 50° C., it will be found to have risen three and a half times higher than the water. Any mixture of alcohol and water, on being treated in the same way, will be found to have a mean point of dilatation between these two, and will be nearer the one or the other, according as either liquid preponderates. If, therefore, a series of mixtures of alcohol and water be made, beginning with—

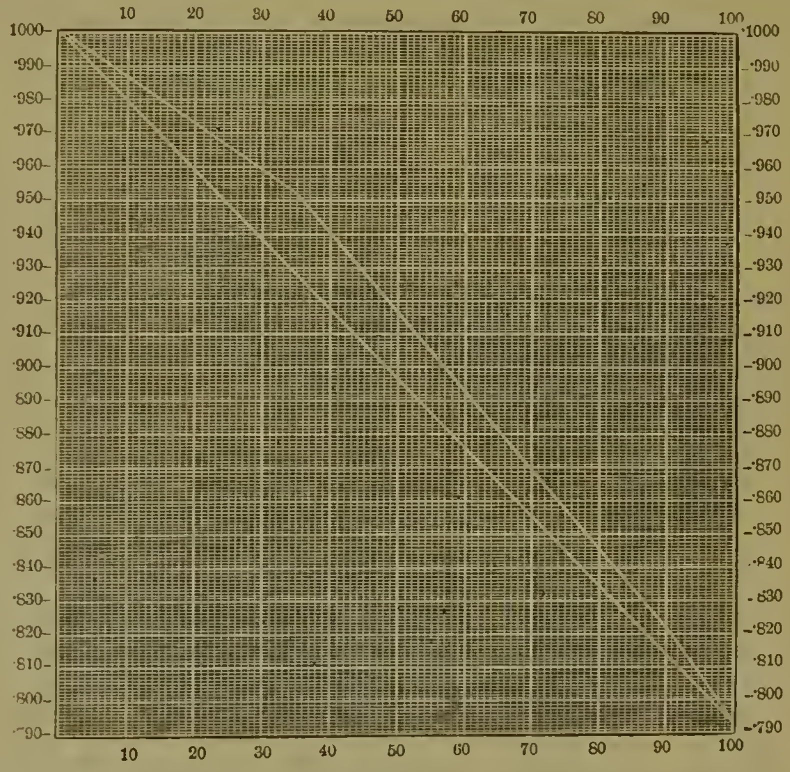
Water	100 parts, ...	Alcohol	0 parts ;
do.	99 "	do.	1 "
do.	98 "	do.	2 "

and so on up to pure alcohol; and their several points of elevation at the respective temperatures (25° to 50° C.), be carefully marked on the tube, a complete centesimal alcoholometric scale will be produced, which will indicate the quantity of alcohol contained in any mixture of alcohol and water, by introducing it at 25° C. and heating it to 50° C.

The same process may be employed with regard to any other two liquids having points of dilatation differing between those of alcohol and water; but it will be understood that the same scale will not serve for more than one mixture. To adapt this principle to the ordinary purposes of alcoholometric measurement, Silbermann constructed a thermometer in a peculiar manner, thereby forming an instrument which he named a *dilatometer*, Fig 53.

Fig. 52.

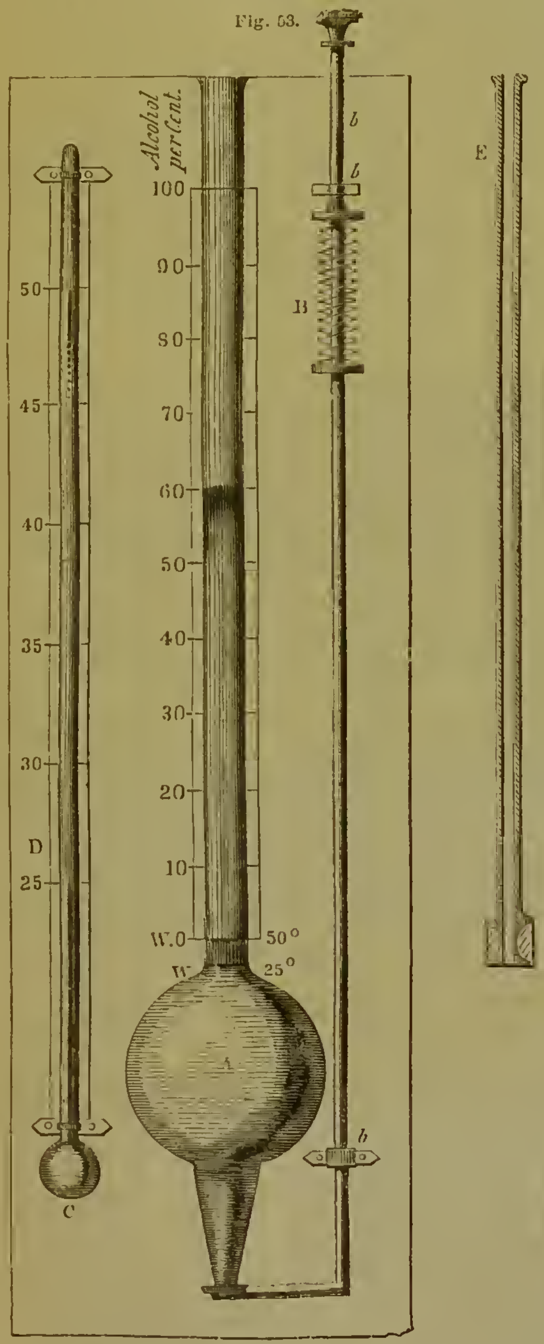
COMPARISON OF MEAN AND ACTUAL SPECIFIC GRAVITIES OF VARIOUS MIXTURES OF ALCOHOL AND WATER



The form and arrangement of this apparatus are these:—A large bulb tube, A, of the form of a hydrometer, but tapering at the bottom and open at the top, is fixed to a metal plate, to which a thermometer, C, is also attached. The thermometer is graduated from 77° to 122° Fahr. (25° to 50° C.). Both the thermometer and bulb of the apparatus, A, are immersed in the liquor to be tested; the former to show the temperature, and the latter to ascertain the expansion of the liquid when it is heated from 25° to 50° C. The expansion of distilled water between the extreme degrees of the thermometer is marked on the stem of A by the figures 25 and 50; from the latter the scale of degrees from 1 to 100 appended to the stem of the apparatus commences, and by it the expansion of the alcoholic liquors is ascertained, when such liquors are heated from 25° to 50° C. A valve of cork or india-rubber closes the tapering end of A; this valve is attached to a rod, *b b*, fastened to the supporting plate and connected to a spring, B. To cause the liquid under examination to flow, the spring valve is depressed by turning a screw of four threads for the purpose of giving a quick motion, fitting in the upper part of the rod, and by reversing the motion of the screw the thermometer is closed; sometimes the rod may be made to terminate in a flattened end or cap, as the figure represents, and is moved by pressing it with the finger. As the liquids are often impregnated with air or gas, it is found necessary, before testing, to remove it; the best method of effecting this is by means of a vacuum, which may be produced by the use of a small leather piston, E, working in the tube of the thermometer. This piston serves first, by suction, to fill the thermometer from below; and then, the lower part being closed, by two or three strokes to separate the air from the liquid. To effect the withdrawal of the piston without any shock, so as not to divide the column abruptly, the piston-rod is made hollow throughout; the operator, having wetted his finger, applies it to the top of the piston-rod, in order to create a vacuum as he draws up the piston; he then withdraws it to readmit the air, and the piston is thus removed without a shock.

In order to form the vacuum properly, the liquid must be pumped in until it rises to the piston-rod; on depressing the piston there is no air left underneath it. The tube is now full of liquid, and by depressing the spring valve the liquid is run off, until it is as high as the lowest mark on the tube, when the temperature has been for two or three minutes at the lowest degree of the mercury thermometer.

Fig. 53.



Any salts or vegetable substances present in wines or alcoholic liquors do not materially affect the result. There is no occasion to fear the presence of any liquid more dilatable than alcohol, since such liquids are more expensive, and may, besides, be detected by their peculiar taste and smell. The same reasoning is also applicable to liquids less dilatable than water. The initial temperature, 25° C., was selected because water may always be found below that temperature in summer. The final degree, 50° C., was chosen to avoid the effect of evaporation, which might diminish the actual degree if it approached too near the boiling point; and the range between this and 25° C. was found sufficient. Besides, these two points offer great facilities for the experiment, as, if it be conducted in a vessel containing about a quart of water, a small spirit lamp underneath it will be sufficient to maintain either the one or the other. The plate carrying the thermometers serves to agitate the water, that its temperature may be uniform throughout.

Dicas' hydrometer is used in America for testing distilled liquors; it is made of copper, with a stem pointed on the summit to receive brass poises, and is accompanied by a graduated ivory scale, with a sliding rule and thermometer to make corrections for temperature. By this instrument the strength of the spirit is indicated, as with Sikes' hydrometer, by a certain number above or below proof; it is not much used in this country.

The alcoholometer of Tralles is used in Russia, those of Cartiers and Gay-Lussac in France, all of which determine the percentage by volume of alcohol in a liquid, and by means of the calculation which is given in the foregoing, the percentage by weight is obtained. Having the percentage of alcohol by weight in a liquid, the quantity of water is likewise found by deducting the amount of alcohol from 100; but if the amount of alcohol be determined by volume, this rule will not answer, since, on mixing alcohol and water, a contraction takes place. In the distillation of spirit it is often necessary to reduce stronger alcoholic liquors to lower degrees of strength; and unless the amount of contraction be known, considerable labour will be attendant on bringing the mixture to the desired quality. The following table shows the relative volumes of alcohol and water which, when mixed, make up 100:—

100 Volumes of spirit contain at 59° Fahr. (15° C.).		100 Volumes of spirit contain at 59° Fahr. (15° C.).	
Volume of alcohol.	Volume of water.	Volume of alcohol.	Volume of water.
100	0·00	45	58·64
95	6·18	40	63·44
90	11·94	35	68·14
85	17·47	30	72·72
80	22·87	25	77·24
75	28·19	20	81·72
70	33·14	15	86·20
65	38·615	10	90·72
60	43·73	5	95·31
55	48·77	0	100·00
50	53·745		

The annexed table by Gay-Lussac shows the volume of water per cent. which is to be added to a liquor, of whatever strength, to bring it to any degree of dilution.

In this table the top horizontal column indicates the percentage by volume of the spirit which is required to be produced. The vertical columns under the top line specify the number of volumes of water which are to be added to 1000 volumes of alcohol, the richness of which is pointed out in the vertical column at the left-hand side of the table, in order to obtain the spirit properly diluted.

GAY-LUSSAC'S TABLE FOR DILUTION OF ALCOHOL.

1000 vols. of alcohol of per cent. by vol.	Desired strength of the spirit.																			
	Per cent. by volume.																			
	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49
31	33																			
32	67	32																		
33	100	65	31																	
34	134	97	63	30																
35	167	129	94	61	30															
36	201	162	126	91	59	29														
37	234	194	157	122	89	58	28													
38	268	227	189	153	119	86	56	27												
39	302	260	220	183	148	115	84	55	27											
40	335	292	252	214	178	144	112	82	53	26										
41	369	325	284	245	208	173	140	109	80	52	25									
42	403	358	315	275	238	202	169	137	107	78	51	25								
43	437	390	347	306	268	231	197	164	134	104	76	50	24							
44	471	423	379	337	298	261	225	192	160	130	102	75	49	24						
45	505	456	411	368	328	290	254	220	187	157	127	99	73	47	23					
46	539	489	443	399	358	319	282	247	214	183	153	124	97	71	46	23				
47	573	522	474	430	388	348	310	275	241	209	179	149	122	95	70	46	22			
48	607	555	506	461	418	377	339	303	268	235	204	174	146	119	93	68	45	22		
49	641	588	538	492	448	407	367	330	295	262	230	200	171	143	116	91	67	44	21	
50	675	621	570	523	478	436	396	358	322	288	256	225	195	167	140	114	89	66	43	21
51	709	654	602	554	508	465	424	386	349	314	281	250	220	191	163	137	112	87	64	42
52	743	687	634	585	539	495	453	414	376	341	307	275	244	215	187	160	134	110	86	63
53	777	720	666	616	569	524	482	442	403	367	333	300	269	239	210	183	157	132	107	84
54	811	753	699	647	599	553	510	469	431	394	359	325	293	263	234	206	179	153	129	105
55	846	786	731	679	629	583	539	497	458	420	385	350	318	287	257	229	202	176	151	127
56	880	820	763	700	660	613	568	525	485	447	411	376	343	311	281	252	224	198	172	148
57	914	853	795	741	690	642	596	553	512	473	436	401	367	335	305	275	247	220	194	169
58	919	886	827	772	721	672	625	581	540	500	462	426	392	359	328	298	269	242	216	190
59	983	919	860	804	751	701	654	609	567	527	488	452	417	384	352	321	292	264	237	212
60	1017	953	892	835	781	731	683	637	594	553	514	477	442	408	375	345	315	286	259	233
61	1052	986	924	867	812	760	711	665	622	580	540	503	467	432	399	368	338	309	281	254
62	1086	1019	957	898	842	790	740	694	649	607	566	528	491	456	423	391	360	331	303	276
63	1121	1053	989	929	873	820	769	722	676	633	593	554	516	481	447	414	383	353	325	297
64	1155	1086	1022	961	904	850	798	750	704	660	619	579	541	505	471	438	406	376	346	318
65	1190	1120	1054	992	934	879	827	778	731	687	645	605	566	529	494	461	429	398	368	340
66	1224	1153	1086	1024	965	909	856	806	759	714	671	630	591	554	518	484	451	420	390	361
67	1259	1187	1119	1055	995	939	885	834	786	741	697	656	616	578	542	508	474	443	412	383
68	1293	1220	1151	1087	1026	969	914	863	814	767	723	681	641	603	566	531	497	465	434	404
69	1328	1254	1184	1118	1056	998	943	891	841	794	750	707	666	627	590	554	520	487	456	426
70	1363	1287	1216	1150	1087	1028	972	919	869	821	776	732	691	652	614	578	543	510	478	447
71	1397	1321	1249	1182	1118	1058	1001	948	897	848	802	758	716	676	638	601	566	532	500	469
72	1432	1354	1282	1213	1149	1088	1030	977	924	875	828	784	741	701	662	625	589	555	522	491
73	1467	1388	1314	1245	1180	1118	1060	1005	952	902	855	810	767	725	686	648	612	578	544	512
74	1502	1422	1347	1277	1211	1148	1089	1033	980	929	881	835	792	750	710	672	635	600	567	534
75	1536	1456	1380	1309	1241	1178	1118	1061	1008	956	908	861	817	775	734	695	658	623	589	556
76	1571	1489	1413	1340	1272	1208	1147	1089	1035	983	934	887	842	799	758	719	681	645	611	578
77	1606	1523	1445	1372	1303	1238	1177	1118	1063	1011	961	913	867	824	782	743	705	668	633	599
78	1641	1557	1478	1404	1334	1268	1206	1147	1091	1038	987	939	893	849	807	766	728	691	655	621
79	1676	1591	1511	1436	1365	1299	1235	1175	1119	1065	1014	965	918	873	831	790	751	713	678	643
80	1711	1625	1544	1468	1396	1329	1265	1201	1147	1092	1040	991	943	898	855	813	774	736	700	665
81	1746	1658	1577	1500	1427	1359	1294	1233	1175	1119	1067	1017	969	923	879	837	797	759	722	687
82	1781	1692	1610	1532	1458	1389	1323	1261	1203	1147	1093	1043	994	948	904	861	821	782	745	709
83	1816	1726	1643	1564	1489	1419	1353	1290	1231	1174	1120	1069	1020	973	928	885	844	805	767	731
84	1851	1760	1676	1596	1521	1450	1382	1319	1259	1201	1147	1095	1045	998	952	909	867	828	789	753
85	1886	1794	1709	1628	1552	1480	1412	1348	1287	1229	1173	1121	1071	1023	977	933	891	851	812	775
86	1921	1828	1742	1660	1583	1510	1442	1376	1315	1256	1200	1147	1096	1048	1001	957	914	874	834	797
87	1956	1863	1775	1692	1614	1541	1471	1405	1343	1284	1227	1173	1122	1073	1026	981	938	897	857	819
88	1992	1897	1808	1724	1645	1571	1501	1434	1371	1311	1254	1200	1147	1098	1050	1005	961	920	880	841
89	2027	1931	1841	1757	1677	1602	1531	1463	1400	1339	1281	1226	1173	1123	1075	1029	985	943	902	863
90	2062	1966	1875	1789	1708	1633	1561	1492	1428	1367	1308	1252	1199	1148	1100	1053	1009	966	925	886

GAY-LUSSAC'S TABLE FOR DILUTION OF ALCOHOL.—*Continued.*

1000 vols. of alcohol of per cent. by vol.	Desired strength of the spirit.																								
	Per cent. by volume.																								
	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74
51	21																								
52	41	20																							
53	62	41	20																						
54	83	61	40	19																					
55	103	81	60	39	19																				
56	124	102	80	59	38	19																			
57	145	122	100	78	58	38	19																		
58	166	142	120	99	77	57	37	18																	
59	187	163	140	118	96	76	56	37	18																
60	208	183	160	137	116	95	74	55	36	18															
61	229	204	180	157	135	114	93	73	54	35	17														
62	250	225	200	177	155	133	112	92	72	53	35	17													
63	271	245	221	197	174	152	131	110	90	71	52	34	17												
64	292	266	241	217	194	171	150	128	109	89	70	52	34	17											
65	313	286	261	237	213	190	168	147	127	107	88	69	51	33	16										
66	334	307	281	256	233	209	187	166	145	125	105	86	68	50	33	16									
67	355	328	301	276	252	229	206	184	163	143	123	104	85	67	49	32	16								
68	376	348	322	296	272	248	225	203	181	160	140	121	102	84	66	49	32	16							
69	397	369	342	316	291	267	244	221	200	178	158	138	119	101	82	65	48	32	16						
70	418	390	362	336	311	286	263	240	218	196	176	156	136	117	99	81	64	47	31	15					
71	439	411	383	356	331	306	282	259	236	214	193	173	153	131	116	98	80	63	47	31	15				
72	460	431	403	376	350	325	301	277	255	232	211	191	171	151	132	114	97	79	63	46	30	15			
73	482	452	424	396	370	344	320	296	273	251	229	208	188	168	149	131	113	95	78	62	46	30	15		
74	503	473	444	416	390	364	339	315	291	269	247	226	205	185	166	147	129	111	94	77	61	45	30	15	
75	524	494	465	437	409	383	358	333	310	287	265	243	222	202	183	164	145	127	110	93	76	60	45	29	14
76	546	515	485	457	429	403	377	352	328	305	283	261	240	219	199	180	162	143	126	109	92	75	60	44	29
77	567	536	506	477	449	422	396	371	347	323	300	278	257	236	216	197	178	159	142	124	107	91	75	59	44
78	588	557	527	497	469	442	415	390	365	341	318	296	274	253	233	213	194	176	157	140	123	106	90	74	58
79	610	578	547	517	489	461	434	409	384	360	336	314	292	271	250	230	211	192	173	155	138	121	105	88	73
80	631	599	568	538	509	481	454	428	402	378	354	331	309	288	267	247	227	208	189	171	153	136	120	103	87
81	653	620	588	558	529	500	473	447	421	396	372	349	327	305	284	263	243	224	205	187	169	152	135	118	102
82	674	641	609	578	549	520	492	465	440	415	390	367	344	322	301	280	260	240	221	203	184	167	150	133	117
83	696	662	630	599	569	540	512	485	458	433	409	385	362	339	318	297	276	256	237	218	200	182	165	148	131
84	717	683	651	619	589	559	531	504	477	451	427	403	379	357	335	313	293	273	253	234	216	198	180	163	146
85	739	705	671	640	609	579	550	523	496	470	445	421	397	374	352	330	309	289	269	250	231	213	195	178	161
86	761	726	692	660	629	599	570	542	515	488	463	438	415	391	369	347	326	305	285	266	247	229	211	193	176
87	782	747	713	681	649	619	589	561	534	507	481	456	432	409	386	364	343	322	302	282	263	244	226	208	191
88	804	769	734	701	669	639	609	580	553	526	500	474	450	426	403	381	359	338	318	298	279	260	241	223	206
89	826	790	755	722	690	659	629	600	572	544	518	493	468	444	421	398	376	355	334	314	295	275	257	239	221
90	848	812	777	743	710	679	648	619	591	563	537	511	486	462	438	415	393	372	351	331	311	291	273	254	236

GAY-LUSSAC'S DILUTION TABLE.—*Concluded.*

1000 vols. of alcohol of per cent. by vol.	Desired strength of the spirit.														
	Per cent. by volume.														
	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89
76	14														
77	29	14													
78	43	28	14												
79	57	43	28	14											
80	72	57	42	28	14										
81	86	71	56	42	27	14									
82	101	85	70	56	41	27	13								
83	116	100	85	70	55	41	27	13							
84	130	114	99	84	69	55	40	27	13						
85	145	129	113	98	83	68	54	40	26	13					
86	159	143	127	112	97	82	68	54	40	26	13				
87	174	158	142	126	111	96	81	67	53	39	26	13			
88	189	172	156	140	125	110	95	81	66	53	39	26	13		
89	204	187	171	155	139	124	109	94	80	66	52	39	26	13	
90	219	202	185	169	153	138	123	108	94	79	66	52	39	26	13

As an example. If it be required to prepare an alcohol of 50 per cent. from a liquor which is 80 per cent., the number, 50, is sought in the top horizontal line of strengths, and in the vertical line under this figure the number 631 is found in a horizontal line with 80 in the left-hand vertical column; this figure indicates the number of volumes of water which are to be added to 1000 volumes of alcohol of 80 per cent. to bring it to the required degree of dilution.

Again, if it be requisite to bring a spirit of 60 per cent. to 30 per cent., the latter figure is found in the horizontal column at the top, and in the vertical column under it, in a line with 60 on the left hand, 1017 is seen, which is the number of measures of water required to reduce 1000 volumes of the strong liquor to the strength of 30 per cent.

The tables for determining the percentage of alcohol, as given in the preceding pages, serve to give a clear view of the composition of alcoholic liquors of different states of dilution, and under the influence of various degrees of temperature and pressure, enabling the diligent student to draw up tables of great accuracy and of less extent for reference on all ordinary occasions. From what has been said upon the properties of alcohol, in relation to its real specific gravity, it will be observed that none of the enumerated tables are strictly correct, since the density of absolute alcohol—to which all the other densities bear a proportionate analogy—stated in them, is considerably higher than that assigned to it by chemists.

The annexed is a contraction of the foregoing table of GAY-LUSSAC, showing the dilution per cent. in reducing liquors to a lower strength. The upper horizontal column contains the per cent. of the stronger alcohol, and the vertical columns below, the bulk of water which is to be added to 100 volumes of it, to produce spirit of the quality indicated in the left-hand column.

Desired strength in per cent.	100 vols. of alcohol of per cent. by vol.								
	90	85	80	75	70	65	60	55	50
85	6·56								
80	13·79	6·83							
75	21·89	14·48	7·20						
70	31·05	23·14	15·35	7·64					
65	41·53	33·03	24·66	16·37	8·15				
60	53·65	44·48	35·44	26·47	17·58	8·76			
55	67·87	57·90	48·07	38·32	28·63	19·02	9·47		
50	84·71	73·90	63·04	52·43	41·73	31·25	20·47	10·35	
45	105·34	93·30	81·38	69·54	57·78	46·09	34·46	22·90	11·41
40	130·80	117·34	104·01	90·76	77·58	64·48	51·43	38·46	25·55
35	163·28	148·01	132·88	117·82	102·84	87·93	73·08	58·31	43·59
30	206·22	188·57	171·05	153·61	136·04	118·94	101·71	84·54	67·45
25	266·12	245·15	224·30	203·53	182·83	162·21	141·65	121·16	100·73
20	355·80	329·84	304·01	278·26	252·58	226·98	201·43	175·96	150·55
15	505·27	471·00	436·85	402·81	368·83	334·91	301·07	267·29	233·64
10	804·54	753·65	702·89	652·21	601·60	551·06	500·59	450·19	399·85

Adulterations of Spirituous Liquors and their Detection.—The sophistication of alcoholic liquors has been practised to give to many solutions containing very little spirit, the appearance and some of the physical properties which would be conferred by alcohol; and adulterations have also been practised with a view, as it were, of veiling the quantity of spirit.

The counterfeiting of strong alcoholic liquors apparently as weaker spirit, or passing them off as quite a different liquid, was fostered in the wine-growing countries of the Continent, where large quantities of brandy were manufactured; and before the proper means of detection were at command the strongest brandy was often sold to the merchants disguised as ordinary wine, or a much weaker spirit, and by this means the *Octroi* duties were eluded. The imposition had long been practised after the introduction of various hydrometric or alcoholometric scales for the determination of the amount of alcohol in spirituous liquors; for as most of these were on the principle of the gravity test, various substances were added which would heighten the density, and make it appear that the spirit was much more aqueous than it really was. In other instances the spirit was passed upon the excise as quite a different article, sometimes as a deodorising agent, while at other times it was made to assume the character of wood naphtha. Expedients such as these have been practised in this country; and in large towns where much alcoholic liquor is consumed, drinks are frequently sold to the public which are said to be alcoholic, but in reality contain little or no spirit. To defraud the excise, sugar or other extractive matter is added to the spirit to increase the specific gravity, and sometimes wood-spirit, turpentine, pyroligneous acid, chlorine water, or other bodies possessing a strong odour, are added to the liquor, and in this case it is represented as not being alcoholic.

The detection of these frauds is, for the most part, easy and expeditious. When sugar, extractive matter, pyroligneous acid, turpentine, &c., are suspected, it is only necessary to distil a portion of the liquid, and determine the density of the distillate by the hydrometer, or specific gravity bottle, and on referring to the corresponding gravity in either of the tables already given, under *alcoholometry*, the corresponding value of alcohol is found. When a liquor is disguised as wood naphtha, it is more difficult to determine the amount of alcohol; distillation is in this case ineffectual, since the naphtha passes over at even a lower temperature than the alcohol. The following is the method recommended by URE for the detection of alcohol in wood or coal naphtha, or pyroligneous acid:—

A small quantity of nitric acid of specific gravity 1·45 is first to be added to the spirit under examination, and if alcohol be present it will immediately produce an effervescence of nitrous ether (ethyl nitrite, C_2H_5,NO_2), which may be recognized as such by its odour. The mixture is then treated with a solution of mercury in nitric acid—which is prepared by dissolving 100 grains of mercury in 1 fluid ounce of acid, with the help of heat. Soon after this addition, and especially on raising the temperature, the mixture begins to effervesce and to evolve thick ethereal vapours; should the effervescence become too violent, it must be quelled by immediately withdrawing the fire, and cooling the vessel. A yellowish grey precipitate falls down, which is mercuric fulminate ($HgC_2N_2O_2$), and which should be immediately separated by decanting or filtering the liquor from it, washing the precipitate on the filter with a little distilled water, and carefully drying it at a heat which must not exceed 100° Fahr. (37°·7 C.); after which it is weighed. The quantity of mercuric fulminate obtained is nearly equal to that of the alcohol contained in the wood-spirit; and at any rate, the formation of the detonating salt is quite characteristic of the presence of alcohol, since wood-spirit treated by nitric acid in the presence of mercury or silver produces neither fulminate of silver nor fulminate of mercury.

In applying this test the greatest care should be exercised, and every precaution taken to prevent contact of any hard body with the precipitate, since the fulminates of silver and mercury, and especially the former, are very explosive; silver fulminate ($Ag_2C_2N_2O_2$) has been known to explode by contact with a glass rod, even under water; the mercuric fulminate is less explosive, and is on that account preferred. For the purpose of collecting these compounds, the feather of a quill should be used; and if the quantity exceeds a few grains, it should be collected in several filters so as to handle only small portions at a time. During the evolution of the ethereal vapours all approach of flame should be carefully avoided.

The same chemist gives the following as the principal tests whereby to discern alcohol from wood naphtha, and also whether the latter is genuine or mixed with alcohol:—

1st. The boiling point of pure wood naphtha spirit is at least 20° Fahr. (5°·5 C.) below that of alcohol of the same density, and it exhales the characteristic, pungent, and offensive odour of aldehyde. In the course of his experiments he found the boiling point of—

	Fahr.	C.
Wood-spirit, of specific gravity ·870, to be	144°	62°·2
Alcohol, of like gravity,................	180°	82°·2
Wood-spirit, of specific gravity ·832,.....	140°	60°·0
Alcohol, of the same density,............	171°·1	77°·2

If 10 per cent. of naphtha be mixed with alcohol, the boiling point is lowered at least 6° Fahr. (3°·3 C.)

2nd. When rectified naphtha is distilled at the temperature of boiling water from a large quantity of lime, the distillate is unchanged in its gravity, whilst if alcohol, or a mixture of alcohol and naphtha, be subjected to the same treatment, the first portions that come over are nearly absolute, and stand at a density below ·800, and contain, at 60° Fahr. (15°·5 C.), about 70 per cent. over proof. The reason of this characteristic difference seems to be that naphtha possesses a stronger affinity for water than alcohol.

3rd. When water is added to alcohol, the specific gravity of the liquor becomes reduced in a less proportion than when wood-spirit of the same gravity as the alcohol is diluted with the same quantity of water. Thus, for example, if alcohol of a given density is diluted with a certain quantity of water, so as to bring it to specific gravity 0·920, wood-spirit of the same original gravity, and diluted with the same quantity of water, will become of specific gravity 0·926 or 0·927.

When brandies, gins, and other alcoholic liquors are artificially made by admixture of various ingredients with alcohol, as is directed under these heads, it is not uncommon to find that the alcohol, in such compounded articles, is attenuated as low as possible, and that the agreeableness of taste and the pungency of flavour peculiar to the genuine beverages, are conferred upon the mixture by means of pepper, cayenne, or other acrid substances. These sophistications are discovered by evaporating a known quantity of the liquid to dryness at a gentle heat, when the added substances will be found as a residue.

Acetates of copper and lead are rarely found in brandies, owing to the repeated distillations to which the spirit is subjected, but when the old stills are employed, the solder connecting the seams of the still is apt to be dissolved by the small quantity of acetic acid present in the liquors; plumbic acetate (sugar of lead) has likewise been known to be added to facilitate the clarifying process. Liquors thus treated are extremely pernicious, since the acetates of lead are highly poisonous. By filtering the brandy through animal charcoal, and adding sulphuric acid to the clear liquor, if the lead is present in excess a white precipitate appears; if there is no precipitate, a stream of sulphuretted hydrogen is to be transmitted through the liquid, and if any lead is present, a black precipitate or coloration is produced. Should a white precipitate be obtained by the action of sulphuric acid or sodium sulphate on the brandy, it is turned black by the addition of ammonium sulphide; and if the precipitate should be bulky it may be mixed with a little sodium carbonate, and reduced on charcoal before the blowpipe to a globule of metal.

Copper may be detected by filtering a portion of the brandy through animal charcoal, to decolorize it; ammonia is then added to the clear liquor, to which it will impart a blue tinge, if copper be present in sufficient quantity. Several hours are sometimes requisite to determine this appearance. The presence of copper may also be detected by immersing a blade of perfectly clean iron in the brandy, and leaving it there for a few hours, when it will be found coated with a film of metallic copper. The brandy, first decolorized by animal charcoal, may also be tested for copper by a solution of potassium ferrocyanide, which will produce a reddish brown precipitate with this metal.

ALUM.—*Alun*, French; *alaun*, German; *alumen*, Latin. According to BECKMAN alum was first discovered in Asia, and was certainly not known to the Greeks or Romans, what the latter called *alumen* being green vitriol (ferrous sulphate). The Greeks and Romans mention only natural alum; now alum is very rarely produced spontaneously, and then only as an efflorescence on stones in the neighbourhood of certain mineral waters. Crystals of alum are also occasionally found upon minerals containing much aluminium when they have been long exposed to the weather; but even then they are so small and so much scattered as to require close observation to discover them. The celebrity acquired by *alumen* among the ancients, as a substance extremely useful in dyeing and medicine, was entirely forgotten at the time that modern alum came into use; but it was again revived when it was discovered that alum could be manufactured from aluminous minerals containing sulphur compounds. This circumstance has served in some measure to strengthen the opinion that the alum of the ancients and of the moderns is synonymous, although the former was found and the latter was extracted by a chemical process. Some historians of the fifteenth century speak of the alum works as if the manufacture in Europe was only a revival.

Alum owes the high estimation in which it is held to its value as a mordant in dyeing.

Alum works existed many centuries ago at Roha, or Roccha, in Mesopotamia, whence the old name of Roch alum is applied to this salt. This is the opinion of Leibnitz, who states that *alumen roccæ* was that kind first procured from Rocca, and that the name was subsequently given to every good species of alum. A few are of opinion that alum obtained from alum-stone has been so called to distinguish it from the alum from schists (schist was employed for making alum in the time of Agricola), which usually contains more iron than the former; and others assert that alum acquired the name *rocca* from the aluminous rocks of Tolfa.

At a later date alum was manufactured near Smyrna, and in the fifteenth century there were alum factories in the vicinity of Constantinople, where John di Castro learned his art, as will be hereafter noticed.

The inhabitants of Genoa, and other commercial people of Italy, imported alum from the above places into Western Europe for the use of dyers of red cloth. When these countries were taken by the Turks the Italians sought for and discovered aluminous minerals in their own soil, which caused the abandonment of many of the Turkish alum works.

Ducas describes very minutely the alum works at Foya Nova, near Smyrna. Francis and Griffith state that in Phocis, lying close to Ionia, there is a mountain rich in aluminous mineral. The stones found at the summit are first calcined in the fire, and then reduced to powder by throwing them into water. The moist mass is put into a kettle, a little more water added, and the whole having been made to boil, the powder is lixiviated, the thick part which falls to the bottom in a cake is preserved, and the hard and earthy portions are discarded. The cake is afterwards allowed to dissolve in vessels for four days, at the end of which the alum is found in crystals around their edges, and their bottoms are also covered with the salt; the remaining liquor is poured into a kettle, diluted with water, and more powder added, then boiled as before, and put into proper vessels to crystallize. The alum obtained in this manner is preserved as an article very necessary for dyers. Captains of ships bound from the Levant to Europe consider alum as a very convenient and useful cargo for their vessels.

The alum works near Civita Vecchia are, by Italian historians, asserted to have been the first in Europe; they are the oldest carried on at present. They were founded by John di Castro, who learned the process near Constantinople. He was there trading in Italian cloths and dyestuffs at the time the superb city fell into the hands of the Turks; after this he returned to his own country, and having found, in the neighbourhood of Tolia, a plant which he had observed growing abundantly in the aluminous districts of Asia, he conjectured that the virgin soil might also contain the same salt, and the astringency of its taste proved he was correct. On this discovery factories were immediately erected, the produce of which was sold to the Venetians, the Florentines, and the Genoese. The stones were first calcined, a large quantity of water was then thrown over them, and when they were entirely dissolved the lie was boiled in great leaden caldrons; after which it was run into wooden vats and allowed to evaporate spontaneously, the result being exceedingly good alum. Pope Pius II. employed more than 800 persons in preparing it. The plant which first induced Castro to search for alum was the prickly evergreen, *Ilex aquifolium*, which in Italy is still considered as an indication that the regions where it thrives abound with alum. This shrub is, however, frequently found growing where there is not the slightest trace of this salt.

It appears, from all that can be learned, that the art of boiling alum was first understood in Italy, but not previous to 1548. The great revenue which the Apostolic Chamber derived from alum induced many to seek aluminous minerals, and factories were built wherever such were found. Alum works soon appeared in Germany; and in 1554 at Oberkaufungen, in Hesse, a factory was in full operation. In 1566 letters patent were granted in England to Cornelius de Vos, a Dutchman, for making "allom and copperas." A bill passed the House of Commons confirming this grant, which it appeared De Vos had assigned to Lord Mountjoye. Nothing is known of his or Lord Mountjoye's subsequent proceedings. Shortly after this date, however, an alum factory was erected at Gisborough, in Yorkshire, in the reign of Queen Elizabeth, by Sir Thomas Chaloner, who, observing the trees tinged with an unusual colour, made him suspicious of its being owing to some mineral in the neighbourhood. He ascertained that the strata abounded with an aluminous salt. At that time the English being strangers to the methods of managing it, there is a tradition that Sir Thomas was obliged to seduce some workmen from the Pope's alum works, then the greatest in Europe.

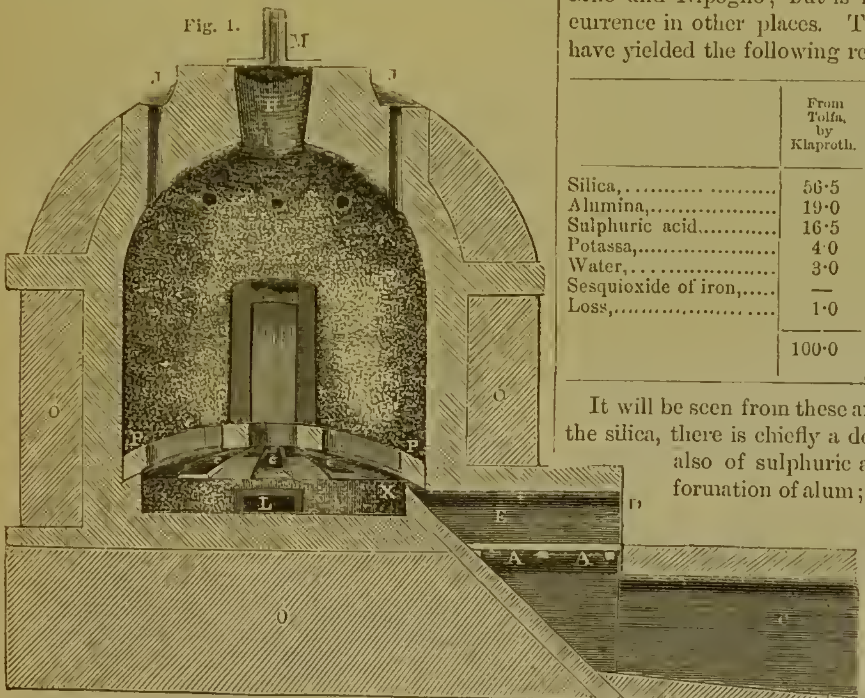
Before entering minutely into the fabrication of alum, it will be proper to state how and where the salt is obtained. The greater portion of the alum in this country is manufactured from alum shale, a bituminous slaty clay containing iron bisulphide diffused in very fine particles throughout its mass; it has a bluish or greenish-black colour, and eliminates sulphurous acid when burned, acquiring thereby an aluminous taste. Many of the alum slates crumble to pieces, or suffer disintegration, on exposure to the air; their sulphur becomes gradually converted into sulphuric acid by absorbing oxygen from the atmosphere, while, simultaneously, the iron is oxidized, and ferrous sulphate and aluminium sulphate are produced. These are separated from the rest of the mass by lixiviation with water. The solution is then concentrated and potassium chloride added. The iron and potassium salts are transformed into ferrous chloride and potassium sulphate, and the latter combining with the aluminium sulphate, constitutes the alum of commerce.

Alum may be prepared by decomposing clay with sulphuric acid. The decomposition is effected by calcining pure clay, grinding the mass to powder, and mixing it with about a half per cent. of sulphuric acid. This mixture is then to be heated in a furnace till the mass becomes very thick; afterwards left to repose for a month or more, and then lixiviated with water to wash out the aluminium sulphate. The addition of potassium sulphate converts it into alum.

Manufacture of Alum.—Two alums only are applied in the arts; these are composed of aluminium sulphate, $Al_2(SO_4)_3$, in combination with potassium sulphate or ammonium sulphate, thus:—Potassio-aluminic sulphate, $AlK(SO_4)_2 12H_2O$; ammonio-aluminic sulphate, $Al(NH_4),(SO_4)_2 12HO$.

The first of these, potash alum, is by practical men simply called alum, whilst the second variety is distinguished by the name of *ammonia alum*. The latter is easily distinguished from the former by its giving off ammonia when triturated with quicklime. Potash alum is preferred by turkey-red dyers; but both are used by dyers and calico-printers solely for their alumina, and in this particular they are accounted nearly of equal value; the other constituents being almost useless.

Fig. 1.



The property which alum possesses, of separating from its concentrated solutions in large, well-defined crystals; the purity of the alumina in this salt, and which is necessary in the applications of alum, enables the purchaser to pay for the sulphuric acid, water, and alkali, although they are useless except for effecting the crystallization. Aluminium sulphate is only with great difficulty separated from the many other extraneous salts (particularly ferrous sulphate) that accompany it in the manufacture; whilst alum, from the ease with which it dissolves in hot water, and its sparing solubility in cold, is readily separable from any adventitious substances.

Production of Alum from Alum Stone.—Alum is obtained in much larger quantity from alum rock, a formation of volcanic origin, than from any other source. This is a massive, granular, only partially crystalline, transparent, and not homogeneous rock, which frequently incloses quartz, sometimes iron pyrites and manganese ore. Its colour is yellowish, passing into green, grey, red, or brown. The pure mineral alum stone, *alunite*, sometimes occurs in it in distinct crystals, which have been found to consist of a basic aluminium sulphate, with potassium sulphate, and is, therefore, a basic alum, or, more probably, a combination of neutral potassio-aluminic sulphate, with aluminium hydrate, $AlK(SO_4)_2Al_2O_3,H_2O$.—*Rammelsberg*. It differs, therefore, from alum in containing an excess of alumina. Alum rock is a massive alunite, but of a less pure character; it is found at Tolfa, near Civita Vecchia; at Montione, in the Comitats, Beregh, and Zemplin, in Hungary; at Mont d'Or, in France; and in the Greek islands, Milo and Nipoglio; but is not of very common occurrence in other places. The analyses of this rock have yielded the following results:—

	From Tolfa, by Klaproth.	From Beregszaz, by Klaproth.	From Montione, by Descotil.	From Mont d'Or, by Cordler
Silica,......................	56·5	62·3	—	28·4
Alumina,.....................	19·0	17·5	40·0	31·8
Sulphuric acid............	16·5	12·5	35·6	27·0
Potassa,.....................	4·0	1·0	13·8	5·8
Water,.......................	3·0	5·0	10·0	3·7
Sesquioxide of iron,.....	—	—	—	1·4
Loss,.........................	1·0	1·7	0·6	1·9
	100·0	100·0	100·0	100 0

It will be seen from these analyses that, overlooking the silica, there is chiefly a deficiency of potassa, and also of sulphuric acid, in the rock, for the formation of alum; but in the mineral from Tolfa, for instance, there is about 3 per cent. of sulphuric acid and 14 per cent. of alumina more than are requisite to form alum with the 4 per cent. of potassa. The alum stone from Beregszaz also contains an excess of 9 per cent. of acid and 16 per cent. of earth; that from d'Or 6 per cent. of acid and 25 per cent. of alumina. At Tolfa, where the alum stone comes to the surface, a quantity of alum is produced proportionate to the amount of potassa in the rock, and the remainder, particularly the excess of alumina, is separated. This is effected by burning the stones in heaps, or furnaces similar to those used in preparing gypsum—Fig. 1—particular care being taken that the temperature does not rise too high.

The inner chamber of this kiln is divided into two unequal portions by an arch, P P, situated about a foot from the bottom. The upper part, into which the alum rock is introduced, partly through the door, G, and partly through the mouth, H, is provided with eight draught holes, J J, the ninth hole being formed by the tube in the covering plate, M. The lower chamber, or fire-space, is in connection with the fire, E, which is situated in front of the kiln, and which is replenished with fuel through the door, D. The draught-channel, C, terminates in the ash-pit under the grate, A, on which the fire is made. The flame enters at X, below the perforated arch, P, where it is uniformly disseminated over the whole area of the kiln, and passes through the fissures, *e e*, and the mass of materials, in an upward direction, escaping at the apertures, J J. If the heat is not uniform throughout the kiln, the draught-holes, J J, are opened on that side where it is least intense, and where it is desirable to lead the flame, while those on the other side are closed. The aperture, L, is used for clearing the fire-chamber, and is closed, as well as G and H, during the firing. Particular attention is given to the maintenance of a proper temperature. O O represents the exterior walls of the kiln, made of non-conducting materials to secure the retention of the heat.

At a red heat aluminium sulphate is decomposed, yielding partly anhydrous sulphuric acid, and partly oxygen and sulphurous acid. As soon as these vapours appear the burning is stopped, and the mass is transferred to walled cisterns, where it is repeatedly moistened with water, and is allowed to disintegrate for three or four months, at the expiration of which period it is converted into a soft mud, tasting perceptibly of alum, which may then be dissolved out with water. If the alum stone contained an excess of aluminium hydrate this would infallibly react upon the alum, and form with it an insoluble compound containing basic sulphate—the burning expels the water from the hydrate of alumina, and thus renders it chemically inactive; the excess of alumina is thus separated from the compound, which then yields an alum soluble in water. During the evaporation—until the spec. grav. is 1·114 at 113° Fahr. (45° C.)—the lie still holds a fine ferruginous rose-red powder in suspension, which feebly colours the crystals, but is left behind when these are redissolved. The crystals contain potassa, but no ammonia, and are known in commerce under the name of *Roman alum.*

PRODUCTION FROM ALUM ORE.—The production of alum from alum shale and alum earth is systematically carried out or divided into three distinct operations: the production of aluminium sulphate; the addition of sulphate or chloride of the alkali to the concentrated cold solution of the former; and lastly, the purification of the alum by recrystallization.

Alum shale is a kind of clay slate impregnated with sulphide of iron and bituminous matters—allied to real clay slate by its firmness, appearance, slaty structure, and great extent. Although it is not so abundantly disseminated as many other species of rocks and minerals, it forms, nevertheless, beds of considerable extent in many localities, particularly in the Scandinavian peninsula; in Bohemia; in the Hartz, in Upper Bavaria; in Voigtland, in the mountainous districts of the Lower Rhine; in England, near Whitby; in Scotland, at Hurlet and Campsie, near Glasgow; in the Uralian Mountains. It may be also met with in many other districts, but not in sufficient quantity to be available for practical purposes.

The following analyses show the composition of the rocks:—

ALUM SHALE FROM SIEHDA, BY LAMPADIUS.

Aluminium sulphate,	2.68
Potash alum,	0·47
Ferrous sulphate,	0·95
Sulphate of lime,	1·70
Silica,	10·32
Alumina,	9·21
Magnesia,	traces
Sesquioxide of iron,	2·30
Oxide of manganese,	0·31
Sulphur,	7·13
Water,	33·90
Carbon, &c.,	31·03
	100 00

ALUM SHALES, BY G. KERSTEN.

	Hermannsschachte.	Gluckaufgang.	Blucherschachte.
Carbonaceous matters,	41·10	27·92	34·20
Silica,	44·02	51·32	50·21
Sesquioxide of iron,	6·23	8·40	0·42
Alumina,	5·60	7·62	5·21
Magnesia,	0·32	0·26	0·53
Oxide of manganese,	0·12	traces	traces
Sulphur,	1·25	2·89	1·72
Sulphate of lime,	traces	traces	traces
Loss,	1·36	1·59	7·71
	100·00	100·00	100·00

ALUM SHALES, BY ERDMANN.

Soluble in acid.	Garnsdorff.		Wezelstein.	
Iron bisulphide,	7·533		10·166	
Silica,	0·060		0·100	
Sesquioxide of iron,	0·966		2·466	
Alumina,	1·833		3·166	
Lime,	0·400		1·000	
Magnesia,	trace		1·022	
		10·792		17·920
Insoluble in acid.				
Silica,	50·066		52 200	
Alumina,	8·900		17·900	
Sesquioxide of iron,	1·300		3·366	
Magnesia,	1·000		1·133	
Lime,	trace		trace	
Coal,	22·833		0·803	
		84·099		75·402
Loss,		5·109		6·678
		100·000		100·000

Subjoined is the composition of several shales which are sometimes used in the manufacture of alum:—

	Locality unknown. D'Aubisson.	Dunmeniss, Devonshire. Stokes.	Gaggenau, Baden. Holtzmann.	Niedesselten, Nassau. Wimph.	Goslar, Hartz. Frick.	Brenndaff, N. Coblentz. Frick.	Lehsten, Thuringerwald. Frick.	Prague. Plerseld.
Silica,	48·6	59·4	64·34	79·17	60·03	62·83	64·57	67·50
Alumina,	23·5	17·4	23·90	10·42	14·91	17·11	17·30	15·89
Sesquioxide of iron,	11·3	11·6	9·70	6·27	8·94	8·23	7·46	5·85
Oxide of manganese,	0·5	—	—	—	—	—	—	0·08
Lime,	—	2·1	—	—	2·08	0·83	1·16	2·24
Magnesia,	1·6	2·2	—	—	4·22	1·90	2·60	3·67
Potash,	4·7	—	—	—	3·87	4·17	1·99	1·23
Soda,	—	—	—	—	—	—	—	2·11
Strontia,	—	—	—	—	—	—	—	0·30
Oxide of copper,	—	—	—	—	0·28	0·27	0·30	—
Fluoride of calcium,	—	—	—	—	—	—	—	1·13
Phosphoric acid,	—	—	—	—	—	—	—	
Sulphur,	0·1	—	—	—	—	—	—	—
Carbon,	0·3	—	—	—	—	—	—	—
Carbonic acid,	—	—	—	—	5·67	4·66	4·62	—
Water,	7·6	6·4	2·22	2·78				—
Loss,	1·8	0·9	—	1·36	—	—	—	—
	100·0	100·0	100·16	100·00	100·00	100·00	100·00	100·00

The annexed is the composition of some shales from Whitby, in Yorkshire, and Campsie, near Glasgow:—

	Whitby Richardson. Top rock.	Whitby Richardson. Bottom rock.		Campsie. Top rock.	Campsie. Top rock.	Campsie. Bottom rock.
Iron bisulphide,	4·20	8·50	Sulphur,	22·36	23·41	Pyrites, 9·63
			Iron,	18·16	15·04	
Silica,	52·25	51·16		15·40	15·40	0·47
Protoxide of iron,	8·49	6·11		—	—	2·18
Alumina,	18·75	18·30		11·35	11·64	18·91
Lime,	1·25	2·15		1·40	2·22	0·40
Magnesia,	0·91	0·90		0·50	·32	2·17
Oxide of manganese,	traces	traces		0·15	—	0·55
Sulphuric acid,	1·37	2·50		—	—	0·05
Potassa,	0·13	traces		0·90	—	1·26
Soda,	0 20	traces		—	—	0·21
Chlorine,	traces	traces		—	—	—
Coal,	4·97	8·29	Carbon and Loss,	29·78	Carbon, 28·80	8·51
Water,	2·68	2·00		—	—	8·54
Loss,	4·80	8·09		—	3·13	0·59
	100·00	100·00		100 00	99·99	100·00

The Campsie alum ores, especially the upper, contain a large excess of pyrites, yielding of course more sulphuric acid than the alumina can take up, while the lower have a considerable excess of alumina; it is, therefore, the object of the manufacturers of alum to mix these ores in such a manner that the different ingredients may be made available as far as possible.

The composition of the residue from the Campsie ores, after calcination and washing out the alum, is:—

Silica,	38·40
Alumina,	12·70
Sesquioxide of iron,	20·80
Oxide of manganese,	traces
Lime,	2·07
Magnesia,	2·00
Potassa,	1·00
Sulphuric acid,	10 76
Water,	12·27
	100·00

It is well known that alum earth belongs to the more recent deposits occurring below the first strata of the tertiary coal formation, which are of a later period than the chalk. It is a massive but soft pulverizable mass, stratified, but not slaty, and of a dark-brown colour; it occupies basins of variable dimensions, according to the position of the neighbouring rocks. Very large deposits of this formation occur in the valley of the Oder, and are worked at Freienwalde and Muskau. It is no uncommon phenomenon, in the coal formations, for the clay and coal strata to permeate each other in those localities where they meet. These deposits, which frequently cover the coal formation, and are at other parts in alternate layers with the coal, play the part of alum ores, and may be worked for the production of alum when they contain a sufficient quantity of sulphide of iron, which is often the case. To this class belong the Scotch ores. In Upper Silesia it is even found profitable to make the refuse coal, or *brees*, which cannot well be used as fuel, subordinate to the production of alum; these coals leave an aluminous ash, and those which are rich in iron pyrites are only distinguished from a real alum ore by their large excess of combustible matter, and which in this case cannot be turned to account.

Iron pyrites, or the bisulphide of iron, is quite as indispensable to the production of alum as alumina itself. It is disseminated through the alum ores, partly in the well-known brilliant yellow crystals, or crystalline deposits, but chiefly in a very fine state of division, as a dull black powder, somewhat resembling the mass obtained by precipitating a salt of iron by ammonium sulphide. Hence, inasmuch as it was not perceptible to the eye, its presence has not only been overlooked in the following analyses, but actually denied:—

	Alum earth. From Freienwalde. Klaproth.	Alum earth. From Putzberg. Bergmann.
Alumina,	16·00	10·90
Silica,	40·00	45·40
Magnesia,	0·25	—
Sulphur,	2·85	3·94
Carbon,	19·15	5·95
Protoxide of iron,	6·40	5·50
" manganese,	—	0·60
Protosulphate of iron,	1·80	5·73
Sulphate of alumina,	—	1·20
" lime,	1·50	1·71
" potassa,	0·50	1·75
Chloride of potassium,	0·50	0·35
Sulphuric acid,	—	0·47
Water,	10·75	16·60
	100·00	100·00

The sulphur is in combination with the iron, and not, as was formerly supposed, in the free state, or as sulphide of carbon. Although the constituents of alum earth are nearly the same in specimens taken from other localities, the proportions are nevertheless variable, as might have been expected from the mode in which the deposits are formed; so great, indeed, is the difference in this respect, that the examples given above can hardly be viewed as a fair average of these compounds. Pyrites and alum ores owe their property of being rapidly decomposed under the influence of atmospheric air to the fine state of division of the bisulphide, and probably, also, to the occasional presence of the protosulphide of iron. Massive crystalline pyrites is under the same circumstances but very slowly decomposed.

The combined action of air and moisture are essential to effect this change. The decomposition is occasioned by 7 equivalents of oxygen and 8 equivalents of water being taken up by the pyrites, FeS_2, which convert it into ferrous sulphate and sulphuric acid, thus:—

$$FeS_2 + 7O + 8H_2O = FeSO_4,7H_2O + H_2SO_4.$$

The combination of the oxygen with the iron and sulphur is accompanied by a spontaneous rise of temperature which results from the chemical union; as the process proceeds, the iron sulphide is converted as by roasting into monosulphide and sulphur, which immediately burn, the former being resolved into ferrous sulphate as shown above, and the latter into sulphurous acid, which is absorbed by the alumina. The aluminium sulphite thus formed is then by further absorption of atmospheric oxygen, transformed into aluminium sulphate ($Al_2(SO_4)_3$). The alum ore, when first taken from the ridges—calcined heaps—and covered with water (which is removed after a short interval), will be found to have yielded little sulphate of alumina to the solution, but a great deal of sesquisulphate of iron; while, after being allowed to remain for some time over the ore, the solution will be found to contain much sulphate of alumina and protosulphate of iron; alum-makers know that the first washes are not those which afford the most alum, unless the ore has been permitted to remain in the steeps for a length of time; they are also aware that more alum is procured by adding to the lie a portion of *mothers*, which are very rich in sesquisulphate of iron, than is obtained by pure water, *per se*.

The sulphides of iron are, therefore, only necessary to the production of alum as affording sulphuric acid to unite with the alumina. Besides the acid produced by the second portion of sulphur, an additional quantity of sulphuric acid is furnished to the alumina by the decomposition of the green vitriol, the ferrous oxide being speedily converted into ferric oxide (Fe_2O_3) by the oxygen of the air, and precipitated in the form of a basic salt. Potassa, which is never altogether wanting in the alum ores, sets free in a similar manner sulphuric acid by the production of potassio-ferric sulphate (iron alum, $FeK(SO_4)_2 12H_2O$). Ferric sulphate is also decomposed by aluminium hydrate, forming aluminium sulphate; this reaction is, however, very slow, and practically of small account.

Potash-iron alum is also always formed when potassium sulphate and ferric sulphate are together in acid solution, and by spontaneous evaporation crystallizes out in octahedral crystals, which in form and taste are not to be distinguished from common alum. These crystals are generally colourless, but sometimes have an amethyst tint, which arises from their containing a trace of ferric acid.—HEINTZ. Potash-iron alum is not stable at temperatures above 32° Fahr. (0° C.), and is decomposed wholly at 212° Fahr. (100° C.).

Lime in the ores is most prejudicial, since it deprives the aluminium and ferrous sulphates of their sulphuric acid, and thus entirely stops the production of alum; ores containing any considerable quantity of lime cannot consequently be used in this manufacture. Gypsum, however, is always found in the crude lies. Magnesia is no less deleterious to the formation of alum, but the magnesium sulphate—Epsom salt—thus produced is in some of the English alum-works regarded as a most important object. Fresh alum ores contain no soluble salts of aluminium or iron; it is only where air has had access to them, either in the pit or at the surface, that efflorescence in the form of fine needles—feather alum—is observed, and this consists partly of real alum, partly of aluminium sulphate, or of combinations of the latter salt with ferrous oxide, magnesium sulphate, or other salts.

An aluminous iron sulphate occurred abundantly some years ago in the Hurlet and Campsie wrought-out coal beds, which had the following composition:—

	Berthier.	Phillips.	R. D. Thomson.	R. D. Thomson.
Sulphuric acid,	34·40	30·90	35·600	28·635
Ferrous oxide,	12·00	20·70	13·560	19·935
Alumina,	8·80	5·20	7·127	2·850
Magnesia,	0·80	—	—	—
Water,	44·00	43·20	43·713	48·580
	100·00	100·00	100·000	100·000

The composition of this feather alum, or *hair-salt*, as it is sometimes termed, is shown by the subsequent analyses. The term hair-salt, sometimes given to these natural effloresced bodies, belongs properly to the magnesium sulphate often accompanying them.

COMPOSITION OF NATURAL ALUMINIUM SULPHATE—FEATHER ALUM ($Al_2(SO_4)_3,18H_2O$).

	Saldana.	Pasto.	Pyromeni, in Island Milo.	Coquimbo, Chili.	Kolosorak, Bohemia.	Friersdorff, near Bonn.	Potschappel, near Dresden.	Freienwalde.	Ararat.	Andes.	Campsie, Glasgow.
	Columbia.										
Sulphuric acid,........	36·400	35·68	40·31	36·97	35·82	37·380	35·710	35·637	58·58	35·872	40·425
Alumina,............	16·000	14·98	14·98	14·63	15·57	14·867	12·778	11·227	38·75	14·645	10·482
Sesquioxide of iron,....	0·004	—	—	2·58	—	—	—	—	—	0·500	8·530
Protoxide of iron,......	—	—	—	—	—	2·463	0·667	·718	$\left.\begin{matrix}SO_3\\FeO\end{matrix}\right\}$ 2·78	—	—
Protoxide of manganese,	—	—	—	—	—	—	1·018	0·307	—	—	—
Potassa,.............	—	—	0·26	—	—	0·215	0·324	0·430	—	—	1·172
Soda,...............	—	—	1·13	—	—	—	—	—	—	2·262	—
Lime,...............	0·002	—	—	—	—	0·149	0·640	0·449	—	—	—
Magnesia,............	0·004	—	0·85	0·14	—	—	0·273	1·912	—	—	—
Hydrochloric acid,......	—	—	0·40	—	—	—	—	—	—	—	—
Silica,..............	—	—	1·13	1·37	—	—	—	0·430	—	0·100	—
Water,..............	46·600	49·34	40·94	44·64	48·61	45·164	47·022	48·847	—	46·375	36·295
Loss,............ ..	0·990	—	—	—	—	—	1·568	·043	—	0·246	3·096
	100·000	100·00	100·00	100·33	100·00	100·238	100·000	100·000	100·11	100·000	100·000
	Boussingault.		Hartwell.	H. Rose.					Gobel.	T. Thomson.	

The manufacture of alum from alum schists may be distributed under the six following heads:—

Preparation of the alum shale.

Lixiviation of the roasted shale.

Evaporation of the lixivium.

Addition of the saline ingredients to precipitate the alum.

Washing of the aluminous salts.

Crystallization.

Preparation of the Alum Shale.—Roasting or Ustulation.—Some alum slates are of such a nature that, when piled in heaps in the open air, and moistened from time to time, they become hot spontaneously, and by degrees fall into a pulverulent mass, which is fit for lixiviation. The greater part of the ores, however, require the roasting process. By roasting the cohesion of the dense slates is so much impaired that their disintegration becomes more rapid; the decomposition of the pyrites is quickened by the expulsion of a portion of the sulphur; and the sulphate of iron already formed is partly decomposed by the heat, and transfers its sulphuric acid to the clay, producing aluminium sulphate.

Such alum shales as contain too little bitumen, or coal, for the roasting process, must be interstratified with layers of small coal or brushwood. The fuel being kindled, the whole slowly ignites. More rock is piled upon it, until in some instances a vast heap of inflamed material, 100 feet high and 200 feet square, is raised, which continues to burn for months. When the heap is fired with brushwood, the ash, containing potash, gives rise to the formation of small quantities of alum.

Alum is manufactured at Whitby by the combustion of the schists of the upper lias, which contain a quantity of iron pyrites and bituminous or carbonaceous matter. The temperature being regulated and water occasionally supplied, decomposition occurs, producing sulphates of alumina, iron, and magnesia, as follows:—

$$4FeS_2 + Al_2O_3 + MgO + O_{28} + 32H_2O = 4(FeSO_4,7H_2O) + Al_2(SO_4)_3 18H_2O + MgSO_4,7H_2O.$$

In Rhenish Prussia, especially at Salzweiler, roasting is effected by the aid of a stratum of brown coal beneath it, which has continued in a state of restricted combustion ever since 1660, when it was accidentally ignited.

A clay bottom is best adapted for the erection of a heap, as it prevents any of the salts being carried by the moisture into the soil; the heap is also sometimes constructed under a shed. As a general rule, the slower and more uniform the heat during the calcination the better the produce.

The Scotch alum-works are those of Hurlet and Campsie—the former six miles south from Glasgow, the latter about nine miles north of the city, at the foot of the Campsie Hills. The aluminous shale found in this locality is interstratified between the coal bed and the limestone, and is nearly as dark-coloured as the former; it is occasionally found mixed with native crystals of sulphate of iron. After the shale has been exposed to the air for some time, the black colour is replaced by a grey, owing to the action of the air causing it to throw out a white efflorescence of alum.

A striking appearance is presented by the alum-field, which is covered throughout with ridges or elongated mounds of the ore for calcination, or already calcined, and these assume a reddish-brown hue from the effect of the heat. The heaps vary in size, and contain from 6000 to about 20,000 tons each. At one of the works under consideration, there are about 15 of the mounds or ridges of shale, each being 120 to 180 feet in length, with a base of about 20 feet, and a height of 15.

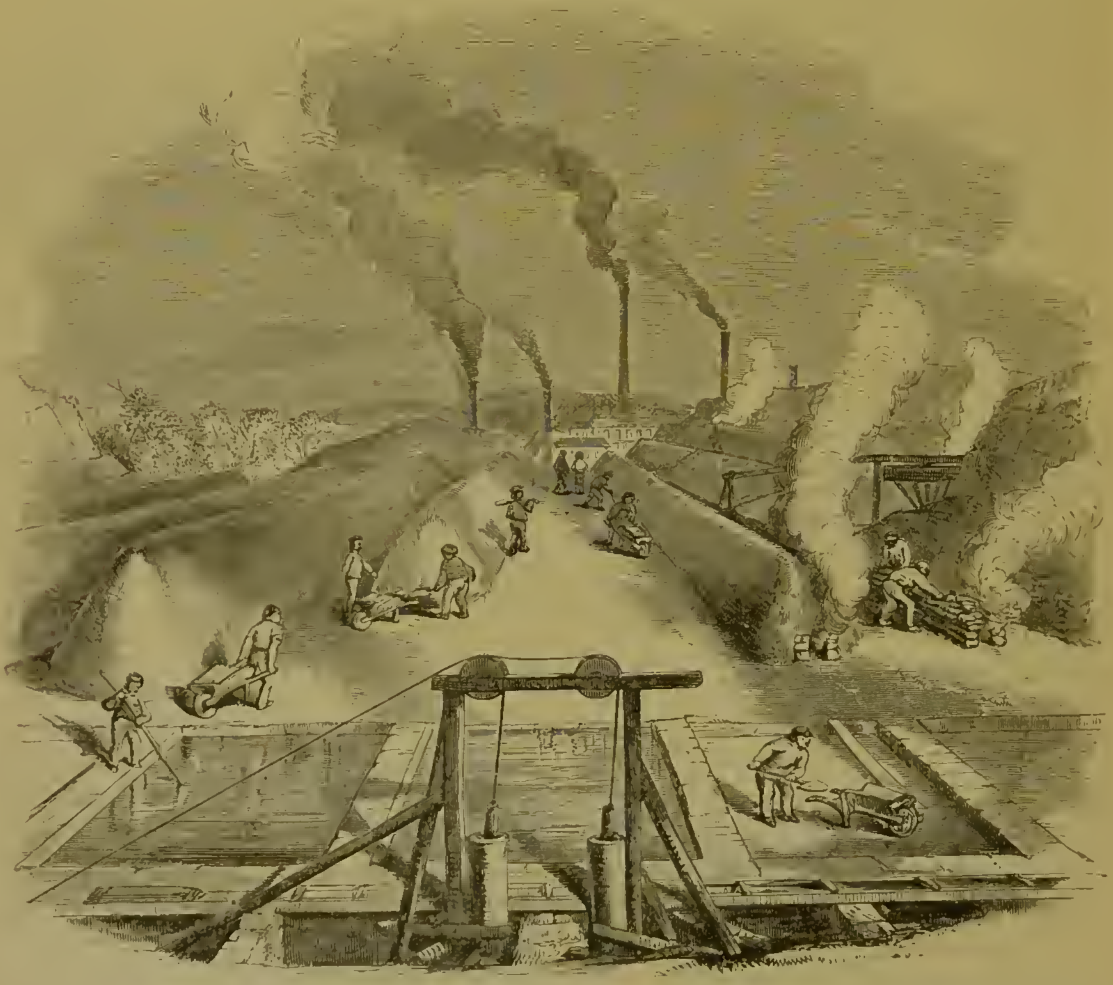
Fig. 2 shows the arrangement of the field.

The mounds are commenced by making a few fires of coal along the intended length, and covering them over with stones or bricks in any convenient manner, leaving lateral ducts, or passages, for the air to enter. The shale is then thrown on the fires, the coarsest first; and as it ignites, and communicates heat to the outer portions, more of the mineral is thrown upon it successively, until the heap is considered large enough, the whole being kept at that state of ignition which practice leads the manager to judge most advantageous. It is then *mantled*, as the workmen term it, that is, covered over with a layer of the already calcined and exhausted ore, in order to protect it from high winds and excessive rains, and also to moderate the heat and let it cool gradually, so that the sulphur present may not be volatilized or sublimed. From three to twelve months, according to the state of the weather, are generally required to calcine the heap properly; in rough weather very little progress is made. This time includes the period of cooling the burned ore, which is done either by leaving the heap to itself, or checking the roasting by applying a thicker mantling.

The several mounds are so arranged that they may be in various stages of advancement, some being cold, others about to be mantled for cooling, many in progress, while a further number are commencing, so that at all times a supply of calcined shale may be ready for the liquefying vats.

At Whitby the calcining heaps are raised from 80 to 100 feet in height. In consequence of the greater amount of carbonaceous matters which the shale at these works contains, the temperature of the mounds sometimes rises too high, and causes the loss of much sulphurous acid; this may be partly prevented by mixing the shale with some of the calcined and exhausted ore, and when the heap has acquired a larger size, if the heat is still deemed too high, either plastering up the crevices with small schist, or mantling over the whole as at Hurlet.

Fig. 2.



About 130 tons of the Whitby calcined schist yield one ton of alum. In this country it is found advisable to pile up on the top of the ridge of brushwood or coal and schist, a pyramidal heap of the mineral, which, having its surface plastered smooth with only a few air-holes, protects the mass from the rains, and at the same time prevents the combustion from becoming too vehement. Should heavy rains supervene, a gutter must be scooped out round the pile for receiving the aluminous mother liquor, and conducting it into the reservoir.

A continuous but very slow heat, with a smothered fire, is most appropriate for the roasting of alum shale. When the fire is too brisk, the iron sulphide sometimes forms with the earthy matters a species of slag, or the sulphur is dissipated in vapour, either of which accidents causes a deficiency in the produce of alum. Those bituminous schists which have been used as fuel under steam boilers have suffered such a violent combustion that their ashes scarcely yield any alum. Even the best regulated calcining piles are apt to burn too briskly in high winds, and under such circumstances should have their draught-holes carefully stopped. It may be laid down as a general rule that the slower the combustion the richer will the roasted ore be in aluminium sulphate. When the calcination is complete the heap is diminished to one half its original bulk; it is covered with a light reddish ash, and is open and porous in the centre, so that the air circulates freely throughout the mass. In order to favour access of air the masses should not be too elevated; and in dry weather a little water should be occasionally sprinkled ou them, which, by dissolving some of the saline matter, renders the interior more open to the atmosphere.

When the calcined mineral is thoroughly cold it is taken to the lixiviating vats. Since many weeks, or even months, may elapse from the first construction of the piles or beds till their complete calcination, care should be taken to provide a sufficient number of them in different stages, so as to have an adequate supply of material for carrying on the lixiviating and crystallizing processes during the course of the year. The complete decomposition of the beds is judged of by the efflorescence of the salt, by the strong aluminous taste of the ashes, and by the appropriate chemical test of lixiviating an aliquot average portion of the mass, and seeing how much alum it will yield with a solution of potassium sulphate or chloride.

Spence calcines the shale by forming on the ground a number of air-channels, on which are laid parallel lines of common bricks at the distance of four inches apart, on these others are placed crosswise; thus the channel formed is about four inches square. The transverse bricks are placed on loosely, so as to allow the air to pass freely upwards; burning coals are laid on the channels, and a layer of the shale which is most bituminous, broken into small pieces; and as the combustion proceeds other layers of the shale fragments, less bituminous than the preceding, are put on continuously, but not in too great a quantity. The thickness of each layer is regulated by the briskness of the combustion, which should never go beyond a low red heat, as a higher temperature would be apt to partially flux the materials, thus rendering the alumina less soluble in the sulphuric acid. The blaze, candle-slates, and other bituminous shales and fire-clay stones, are first broken into lumps about the size of the stones used in road-making. If clay stone be the substance under treatment small coal, or sawdust, must be mixed through it to insure perfect calcination. An examination of the preceding figure, exhibiting the heaps or mounds in various stages of progress, will show that a method similar to this is practised at Hurlet.

The heaps may be made of any convenient dimensions, but the height most appropriate is 4 to 5 feet. The mass will burn out, and be cool enough for use in eight or ten days.

Wilson selects shale as free as possible from impurities, and after exposing it for three or four months to the action of the atmosphere, by which it breaks down and assumes a pulverulent form, roasts it in a lime kiln of the ordinary construction, in which continued removals of roasted shale are effected below, and continued additions of new shale are made in the upper part of the kiln, care being taken to avoid fluxing.

Lixiviation of the Roasted Ores.—This part of the operation for dissolving out the aluminium sulphate and other soluble components, is one which requires much attention. The amount of water must be so regulated as to extract the whole of the soluble bodies, without having a superabundance of liquid to eliminate when the solutions are to be concentrated for precipitation or crystallization. When an excess of water has been employed to exhaust the calcined shale much time and labour are required for the evaporation. Hence, only so much water should be employed as is necessary to exhaust the ore of its soluble saline ingredients. Vessels for the lixiviation are either wooden tanks lined with sheet-lead, or cisterns made of stone; the latter are more durable, but more expensive. The lixiviating vessels are in England and France placed at different levels, for the purpose of facilitating the exhaustion of the material in them, while in Scotland they are constructed upon the same plane. The form usually given to these vessels is that of a square or oblong.

When the exhaustion of the roasted shale is effected by the first of these arrangements, as at Whitby, a wooden or iron sluice carries the lie from the series of tanks to a large cistern to be again returned upon the upper troughs, recharged with fresh portions of the burned ore, in order to bring it to the proper degree of strength, when it is drawn off to be concentrated by heat. Water is let into the upper troughs through several inlets, till it covers the burned material to the depth of one or two inches. After six to twelve hours, according to the facility with which the sulphates are extracted, the liquor from this tank is drawn off at the openings at the bottom into the cistern next beneath it, where it is suffered to remain for an equal length of time before it is run into the clarifying vessel, whence it is conducted to the pans for evaporation. No more than half the water employed in the first washing can be drawn off, as nearly half is retained by the shale; and in consequence of this only half the quantity of water used in the first is added to make the second lie, which, after twelve hours, is drawn off into the lower cistern, like the preceding, and a third quantity of water is poured on in order to extract the whole of the soluble ingredients. This third washing being of a low strength is pumped up to form the first watering of fresh quantities of the material, to bring it to the desired strength for exaporating, which should be from 1·113 to 1·157 specific gravity.

At the Hurlet and Campsie Works, near Glasgow, the process pursued is the following:—After the ore is calcined it is wheeled from the ridges into large stone cisterns, called *steeps*, occupying the same level—see Fig. 2. The steeps are furnished with false bottoms formed of planks, which rest on transverse beams, and the roasted shale is laid on the planks till it is about 18 inches above the bottom. Water, or a weak lie from a previous operation, is poured upon the bed of ore till it is completely covered over, and allowed to remain in contact with it for about eight hours, generally through the night. A plug at the bottom of the vat is then opened, and the solution, which should have a specific gravity of about 20° Twaddle, or 1·100 specific gravity, is drawn off into the *settler*, or supply cistern, where it is allowed to remain at rest, in order that any matters held mechanically may subside. A second wash of a weaker liquor than that employed in the first operation is then added, and left in contact with the partly-exhausted shale as long as in the first instance, after which it is drawn off to the cistern as before; the same operation is repeated a third time if necessary, till the liquid becomes below 12° Twaddle in strength, after which it is not deemed economical to evaporate for alum, the further washings being used for exhausting fresh quantities of ore. The last washings of the steep are made with water, and the strongest of the several resulting weak liquors from each steep is used the first in exhausting a fresh one. After the ore is deprived of its soluble ingredients, it is either removed to the waste heap, or returned to the calcining ridges, where it is intermixed with the fresh ore for the purpose of checking a too rapid combustion, when such is apt to take place.

At Valmunster, in the department of the Moselle, the tanks are constructed of burned stones and clay, and each has a capacity of 1728 cubic feet—1380 cubic feet Rhenish. At the bottom, at certain distances from each other, beams are arranged, upon which a layer of straw and brushwood is laid, and upon this the false bottom of boards is placed. This arrangement forms the filtering apparatus, from which the lie flows through apertures at the sides. Each cistern requires 1280 cubic feet of water for the lixiviation. Lie is first poured upon the ore of 20° B. = sp. gr. 1·157; when this has run off it corresponds to 24° or 25° B., and is fit for boiling; a weaker lie of 15° B. = sp. gr. 1·113, and subsequently others of 10° B., = sp. gr. 1·072, and the weakest of 5° B., = sp. gr. 1·034, then follow; and lastly, the whole is washed out with pure water. All the lies that fall short of 24° or 25° B. are poured upon more or less exhausted ores, according to their strength. By arranging these cisterns upon terraces, the one above the other, the lie can easily be drawn from the upper into the lower cisterns, until it has acquired the proper strength. The solution is then called crude liquor, and is preserved in large walled tanks, ready for further operations.

The density of the liquors is determined by a specific gravity bottle peculiar to the alum manufacturers; it is capable of containing 80 pennyweights of water at the ordinary temperature, and when this bottle is filled with the alum liquor and weighed, the excess of weight is denominated the strength in pennyweights; thus, if a liquor should weigh 90 pennyweights, it would be put as one of 10 pennyweights, or simply liquor of 90. A similar method is the use of the hydrometer, for determining the amount of ingredients dissolved, and this even in the pennyweights of the alum-maker; thus, dividing the indications of Twaddle's hydrometer by 2·5, gives the alum-maker's strength in pennyweights, without the trouble of weighing.

According to SCANLAN, eight different liquors are met with in the alum-works on the Yorkshire coast.

1. *Raw liquor.*—The calcined alum shale is steeped in water till the liquor has acquired a specific gravity of 9 or 10 pennyweights, according to the language of the alum-maker.

2. *Clarified liquor.*—The raw liquor is brought to the boiling point in leaden pans, and suffered to stand in a cistern till it has become clear; it is then called clarified liquor. Its gravity is raised to 10 or 11 pennyweights.

3. *Concentrated liquor.*—Clarified liquor is boiled down to about 20 pennyweights. This is kept merely as a test of the comparative value of the potassium salts used by the alum-maker.

4. *Alum-mother liquor.*—The alum pans are fed with clarified liquor, which is boiled down to about 25 or 30 pennyweights, when a proper quantity of potassium salt in solution is mixed with it, and the whole run into coolers to crystallize. The liquor pumped from these rough crystals is called *alum-mothers.*

5. *Salt-mothers.*—The alum-mothers are boiled down to a crystallizing point, and afford a crop of *rough Epsom*, which is magnesium sulphate with ferrous oxide.

6 and 7. *Alum-washings.*—The rough crystals of alum—No. 4—are washed twice with water, the first washing being about 4 pennyweights, the second about 2½, the difference in gravity being due to mother-liquor clinging to the crystals.

8. *Tun-liquor.*—The washed crystals are now dissolved in boiling water, and run into the roching-tuns—wooden vessels lined with lead—to crystallize. The mother-liquor of the *roch-alum* is called *tun-liquor;* it is of course not quite so pure as a solution of roch-alum in water.

With reference to the exhausted residues, two cases may occur: they have either been rendered perfectly porous by the roasting and decomposition, in which case they will have been completely exhausted, and may be thrown away unless they can be used for covering fresh heaps; or they still contain portions of undecomposed ore, and may then be subjected to a second exposure—as at Buchsweiler, in Alsace—either by themselves or mixed with fresh ore.

The process in many other alum-works, where the ores do not require roasting, is essentially the same as that described above. The lixiviation, however, is then carried on with the heaps themselves, and during the process of decomposition; they are erected for this purpose either upon flat wooden boxes, termed Bühnen, or upon a foundation composed of brick-work or clay, which is completely impervious to the liquor, and where the water which is pumped from time to time upon the heap collects, and is thence conducted to the crude liquor cisterns. The soluble salts are thus removed at certain periods, as they are produced during decomposition. The strength of the crude liquor in the cistern must be regulated by the respective prices of labour and fuel, weak liquor requiring more fuel, and strong liquor more labour; it is never advisable, however, to concentrate the liquor to the degree at which it would be saturated with crystallizable salts, such as green vitriol and sulphate of magnesia, as these would crystallize with the slightest amount of evaporation or rise of temperature before it was desirable they should do so. In general, the density indicated by 20° B., = sp. gr. 1·157, is not exceeded. Simon found the crude liquor from Gleissen, in Neumark, to contain the following ingredients:—

Aluminium sulphate,..................	11·085	= 29·678
Ferrous sulphate,......................	9·773	
Sodium sulphate,.......................	2·035	
Magnesium sulphate,..................	1·754	
Manganese sulphate, ...	0·174	
Potassium sulphate,...................	0·095	
Sulphate of lime,........................	0·120	
Sesquichloride of iron, ...	1·872	
Magnesium chloride,..................	0·334	
Aluminum sesquichloride.	0·419	
Sulphuric acid,..........................	0·563	
Hydrochloric acid, ...	1·454	
Water,........................		70·322
		100 000

These constituents are always accompanied by a certain quantity of ready-formed alum, which may be potash or ammonia alum, or both, if a red heat has not been employed in the manufacture; but if a high temperature has been applied, both are decomposed, leaving only alumina. To the production of the potash alum, the alkali naturally contained in the ores contributes, and still more that which exists in the ash of the wood used as fuel; the production of ammonia is attributable to the nitrogen contained in the coal. During the time that the crude lie is clarifying in the large vats, a chemical decomposition ensues by the action of the air upon the ferrous sulphate, which is not prevented by the state of solution of the salt.

In the foregoing analysis nearly the whole of the oxide of iron is precipitated, and only a very small portion remains in solution. This behaviour is turned to account by mixing the crude liquor with the mud obtained in a subsequent operation. This mud is a kind of basic alum, which is soluble and deficient in a certain quantity of, but does not contain enough sulphuric acid to make alum. The basic sesquisalt of iron is called in German *vitriolschmand*, and is collected from time to time from the bottom of the cisterns, and heated to redness; it then parts with its acid, and the peroxide remains, which is used as a red pigment.

Boiling the Crude Lie.—The further treatment of the lie depends upon the quantity of green vitriol which it contains, and in most cases there is quite as much of this salt as of alum. When this happens the lie is used for the production of both salts, and alum and copperas works are generally carried on conjointly. The process of separating the two salts by crystallization varies much in different manufactories.

When, however, the quantity of ferrous sulphate is too small to admit of being profitably extracted, the liquor is at once evaporated until it has attained the specific gravity 1·40. Basic sulphate of iron is deposited, and the liquor assumes a brick red colour. In order to clear it, it is run off into tanks, and after the suspended matter has deposited is syphoned off and transferred to the precipitation tanks.

When the crude lie is very much charged with the iron salt it is evaporated in pans, into which old iron is thrown. The ferric oxide formed by the action of the air is thereby partly precipitated as a basic salt, and the sulphuric acid set free converts a part of the iron into sulphate, hydrogen gas being at the same time given off. The latter reduces the precipitated ferric sulphate to ferrous sulphate, and prevents its further oxidation during the evaporation.

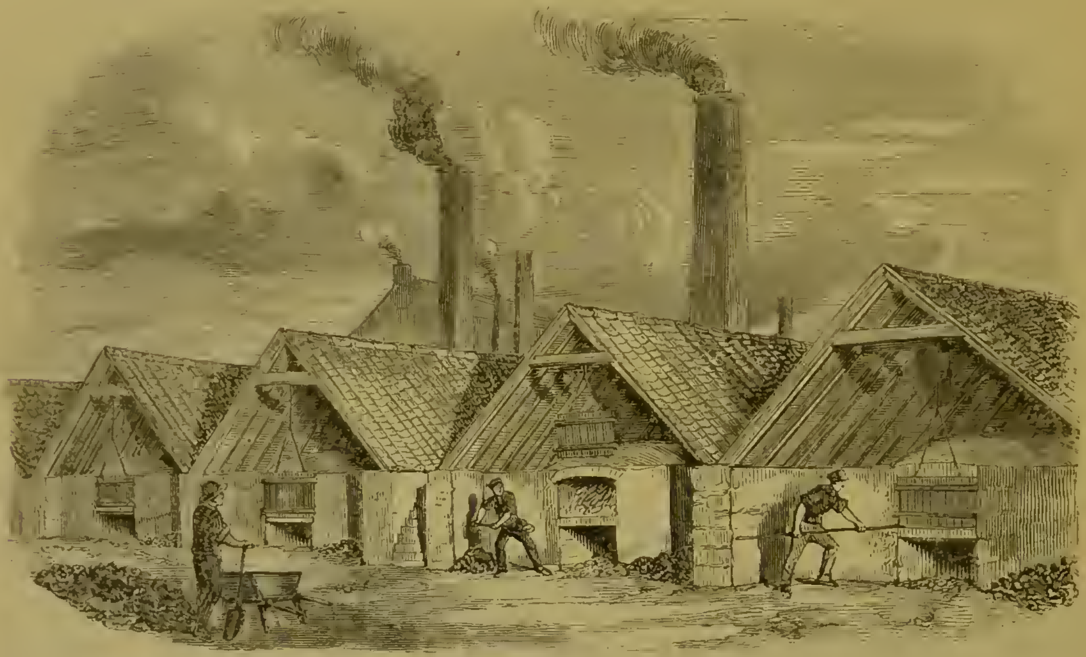
In order to afford more points of attachment for the crystals and facilitate their removal, the workmen hang peeled sticks and branches in the solution. The mother lie contains the whole of the sulphate of alumina, which is separated in a manner to be subsequently described.

In other places—as at Reschwitz, near Saalfeld, at Schwemmsal—the greater portion of the water is removed by causing the liquor to trickle through skeleton towers filled with brushwood placed in a strong draught, as in the salt-works, when, in consequence of the increased action of the air, a considerable portion of the green vitriol is decomposed, and much basic sulphate of iron, mixed with gypsum, is left as an incrustation upon the thorns. The concentrated lie then yields green vitriol on evaporation, and the mother liquor contains the alum.

Another process consists in separating the copperas, not by cooling the saturated solution, but by simple evaporation or abstraction of water. When evaporation has been carried on for some time, and the loss of water and the strength of the lie have reached a certain degree, a point of saturation is attained—when the specific gravity is from 1·35 to 1·37, or 38° to 40° B.—at which the remaining water is just sufficient to retain the copperas in solution. The evaporation of every additional quantity of water then causes the precipitation of a portion of copperas, and a sediment, consisting of very small anhydrous or slightly hydrated crystals, is rapidly deposited. In the meantime, the evaporated water is constantly replaced by fresh liquor. It is obvious that, upon this plan, in which the liquor is always saturated, the quantity of dissolved green vitriol cannot accumulate in the pan, whilst the amount of the other soluble salt, aluminium sulphate, must increase synchronously with the deposition of the former until the liquid is saturated with it. At this stage the liquor is carefully watched by means of the hydrometer, to avoid any supersaturation with aluminic salt, as in time it would precipitate with the ferrous sulphate. The precipitated sulphate of iron is then removed and purified by crystallization, when it acquires seven equivalents of water, and a mother liquid remains, saturated with aluminium sulphate, which is available for the production of alum.

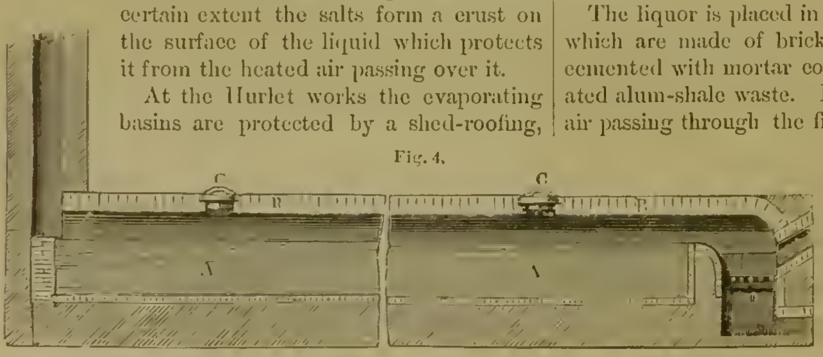
When, on the contrary, the copperas is not in excess, the process is reversed, and begins with the production of alum from the crude liquor. For this purpose the lie is pumped at once from the crude liquor cisterns, in which it has been clarified, into the evaporating pans to be brought to the proper degree of concentration. If the pans are made of tinned iron they will be corroded by the lie, with evolution of hydrogen and the precipitation of basic aluminium sulphate; this action is not prevented by the addition of the basic mud before mentioned. When cast-iron or leaden pans are employed, which are not thus affected, the sediment frequently gets burnt on the bottom, and causes difficulty and damage; but the former are brittle, and the latter are too easily melted by the heat. To prevent this burning metal evaporating pans are now, except under special circumstances, universally discarded, and the flame is carried over the surface of the liquid instead of below the pan. When, however, the liquors contain magnesium sulphate surface evaporation cannot be used, as when the concentration has proceeded to a certain extent the salts form a crust on the surface of the liquid which protects it from the heated air passing over it.

Fig. 3.



At the Hurlet works the evaporating basins are protected by a shed-roofing, as represented in Fig. 3. They are long cisterns of brickwork well cemented together, and arched over with the like materials; they have a length of 60 feet and a breadth of 6, with a depth of about 4; are capable of evaporating about 4500 to 5000 gallons in twenty-four hours. The cisterns are heated by a furnace, which is placed at one end, and on a level almost with the surface of the liquid when the boiler is full, as has been already described; a powerful chimney at the opposite end of the boiler creates a strong draught that sweeps the flame and heat from the fire over the surface of the liquor, producing a very high temperature, and rapid evaporation takes place. Fig. 4 represents a longitudinal section of one of these evaporating cisterns.

Fig. 4.



The liquor is placed in the basin, A A, the walls of which are made of brick, placed upright, and well cemented with mortar composed of lime and lixiviated alum-shale waste. In order that the current of air passing through the fire at D may not carry any appreciable quantity of the ashes into the liquor, the fire bars are fixed low down; the flame passes below the flat arch, B B, to the chimney. This arch contains apertures, C C, through which to observe the different parts of the interior. Fresh liquors, or the mother liquors from a previous operation, are introduced through the openings, C C, as the water is evaporated from the contents of the furnace, until the solution acquires a density of about 1·40, which is the most suitable state of concentration for producing the flour, or meal, for the next operation.

To economize fuel the furnace is very long, and the fire communicates with an evaporating pan, so that the whole heating power may be made available as far as practicable. The draught of the furnace insures, in the simplest manner, a constant current of air over the liquid, which is favourable to evaporation by sweeping the vapours generated onwards to the chimney. During the evaporation of the liquor in the furnace a copious sediment is thrown down, consisting of basic ferric sulphate, sulphate of lime, and other salts, of which it is necessary to clear the liquor in the cooling vats; but however impure the liquor, or however rich in aluminium sulphate the solution may be, its evaporation should not be carried on so far as to cause a deposition of salt upon cooling; the ingredients must be entirely held in the clear solution, even after cooling the liquor in the vats set apart for the production of the alum flour.

The furnaces are fired incessantly for eight days, and the boiler is replenished with liquor from the supply cistern till the eve of the eighth day, when a charge of mother liquor is run in which deposits no sedimentary matter on boiling, like the fresh liquor; this is boiled down till it has the proper degree of strength, and then allowed to settle for twelve hours. After adding the mother liquor it is customary to find the strength of the solution. This is done by taking out a known quantity of it, and concentrating it to the proper strength with potassium chloride and allowing the solution to cool for twenty-four hours; the crystals produced, when washed with a little cold water, dried and weighed, furnish an approximate test of the value of the liquor as to its real percentage of alumina. The alum-maker also employs this process to test the quality of his potassium chloride; but in the latter case he has a standard solution of aluminium sulphate. These experiments point out the quantity of alkaline chloride which should be added per gallon of liquor.

At the Whitby alum-works leaden pans are employed, in consequence of the liquors from the calcined shale of this locality containing sulphate of magnesia, which renders surface evaporation inapplicable, as already explained; the pans are 10 feet long, 4 feet 9 inches wide, 2 feet 2 inches deep at one end, and 2 feet 8 inches at the other. In these lead pans the liquor is rapidly concentrated by a fire at one end, the flue of which runs along in parallel channels under a plate or course of brickwork on which the pans rest. Every morning the pans are emptied into a settling cistern of stone or lead. The specific gravity of the liquor should be about 1·40 to 1·50; this density varies, however, according to the views of the manufacturer. For a liquor which consists of two parts of aluminium sulphate and one of ferrous sulphate, a specific gravity of 1·25 may be sufficient; but for a solution of one part of aluminium sulphate and two parts of sulphate of iron, which latter is to be abstracted by crystallization, a density of 1·40 is requisite.

Preparation of the Powder.—From the evaporating basins the liquor is run into the flour or powder cisterns, in which the aluminium sulphate is combined with potassa or ammonia (the "flux" or "precipitating substance"), in such a manner as to effect the first purification of the alum. The alum is only very slightly soluble in the cold mixture of the liquor with the solution of the precipitating substance, and for this reason the greater portion of it separates in the form of minute crystals, or "flour," from the solution; an agitating motion given to the liquor in the cisterns favours the formation of the crystals. To obtain the largest yield of flour it is necessary for the precipitating solution to be as strong as possible, which is secured by dissolving the precipitant in the least possible quantity of boiling water. The proper amount of the precipitant should be determined, as near as possible, by a preliminary experiment on the small scale; but still these data, though they serve to give an idea of the quantity, do not correspond always with the extensive operations of the manufacturer; so that even when previous experiments are made to ascertain the true quantity to be added, much caution is necessary when operating on the whole bulk of mother liquor, as an excess of the precipitant may often cause much future inconvenience and labour. The best precipitating substances are potassium and ammonium salts, as these form alums which are very insoluble in cold water, only 9·25 parts of the potassium alum, and 9·16 parts of the ammonium alum being dissolved by 100 parts of water at 50° Fahr. (10° C.). Sodium salts are very disadvantageous precipitating agents in the formation of alum, on account of the great solubility of the resulting compound, 46 parts of which are dissolved by 100 of water at 50° Fahr.; they are therefore seldom used.

In the event of potassium salts being employed, preference is given to potassium chloride or sulphate; of the latter 50·9 parts are required to precipitate 100 parts of aluminium sulphate, from which results 334 parts of crystallized alum. The preceding enumerated tests, however, will not answer in the factory, because the mother liquor is indefinite as to the amount of impurities which accompany the aluminium sulphate. To show this more clearly, let it be supposed that the mother liquor is saturated with ferrous sulphate and aluminium sulphate, and that its temperature is 50° Fahr. In this case it will contain, for every

100· parts of water,
33·2 parts of anhydrous ferrous sulphate, and
33·5 parts of anhydrous aluminium sulphate.

To precipitate these products 17·1 parts of neutral potassium sulphate are required, for the solution of which 66 parts of boiling water will be necessary.

On mixing this solution with the lie, 96 parts of alum will be produced, together with the accumulated water of the lie and precipitant, which, collectively, will amount to 166 parts. Of the 96 parts of alum formed, only 16 can remain in solution after the liquor has cooled to the primary temperature of 50° Fahr. (10° C.); therefore 80 parts will separate in the crystallized state; but in practice this quantity diminishes according to the less saturated state of the lie. Since potassium chloride is more soluble in boiling water than potassium sulphate, it is preferable for precipitating the alum flour; the chloride is soluble in 2 parts of boiling and in 3 parts of cold water, and hence the mixture may be made in the cold.

In the instance cited above the 33·5 parts of aluminium sulphate would require 14·5 parts of potassium chloride for precipitation, which would be taken up by 26·2 parts of boiling water, and produce 96·5 parts of alum.

The mixture being made and the precipitate subsided, 12 parts of the alum would be held in solution, so that only 84·5 parts of flour are thrown down in the solid form. If the same quantity of potassium chloride be dissolved in cold water, 46 parts at 50° Fahr. (10° C.) will be required, which, when added to the 23·5 parts of aluminium sulphate in solution, will yield 84 parts of alum as a precipitate, while 13·8 remain dissolved: in both cases the difference is immaterial, whether the precipitant be dissolved in hot or cold water.

Another precipitant which is used with much advantage on account of its great solubility, is ammonium sulphate: this is soluble in 2 parts of water, but the amount of alum obtained is less than when the potassa salts are employed, in the proportion of 100 : 95·6; the reason being that the atomic weight of these bodies differ, the equivalent of ammonium hydrate, $(NH_4)_2$, used being 36, while that of potassium is 39·1.

To recur to the foregoing example, the 33·5 parts of aluminium sulphate will take 15·8 parts of ammonium sulphate to form with them 87·7 parts of alum, of which 77 parts are precipitated and only 10·6 retained in the solution; thus, the disadvantage arising from the lesser equivalent of ammonia alum is counterbalanced by the greater solubility of the ammoniacal salt as a precipitating agent.

The potassium sulphate, for the use of alum-makers, is obtained as a secondary product in the manufacture of nitric acid, and in the purification of potassium hydrate and the crude carbonate of that alkali: thus produced it is an acid sulphate, commonly called bisulphate of potash, the composition of which is expressed by the symbols $KHSO_4$, and cannot be employed for alum making till the excess of acid is neutralized by fresh additions of the caustic or carbonated alkali to form a neutral or bipotassic sulphate of the composition, K_2SO_4.

It is only under the above circumstances that potassium hydrate or carbonate can be employed in the preparation of the alum flour; for if no free acid is present it throws down a basic double sulphate of aluminium and potassium which is insoluble in water, though redissolved by a further addition of bisulphate of potash. A greater or less quantity of this compound is always formed during the evaporation of the liquors, in consequence of the iron present abstracting some of the acid, but, as in the preceding, a slight addition of the bisulphate of potash redissolves it.

Knapp recommends that the purification of crude potassium hydrate and carbonate should be conducted on the same premises with the alum works, for the purpose of turning to use the bisulphates and chloride formed in this operation as precipitants of the alum. With regard to the latter salt, he says it is of greater importance than the sulphate, inasmuch as it effects the decomposition of the sulphates of iron, giving rise to potassium sulphate and chlorides of iron, which are highly deliquescent salts, and from which the alum flour can be removed with greater ease than from the sulphates of iron. But the decided advantage attending the use of the chloride is when a ferric sulphate is present in the lie, as this latter salt, in combination with potassium sulphate, forms a difficultly soluble double salt (basic iron alum), which precipitates in the form of a yellow powder, and contaminates the alum flour. As an excess of potassium chloride would convert the aluminium sulphate into aluminium chloride, a result which would entail a loss, he recommends that the lie should be examined before each operation, with a view to determine the proper amount of chloride to be added, as it happens that the quantity required to precipitate the whole of the aluminium as alum seldom or never meets with sufficient ferrous sulphate in the lie to be converted into potassium sulphate. This examination is conducted as follows:—A saturated solution of the precipitating salt is added from a burette, or other graduated vessel, to a measured quantity of the liquor, the mixture being briskly agitated at each addition; successive portions of the reagent are poured in as long as the flour thrown down increases in quantity, and when no further precipitation is observed the number of measures is read off, which, with sufficient accuracy on the large scale, will stand for the amount of solution required to precipitate a certain amount of the liquor; by this means the tedious process of weighing is avoided. If potassium sulphate has been employed, from its difficult solubility in the cold it may precipitate during the cooling of the liquor, apparently increasing the bulk of the precipitate, and giving rise to an error in the calculation. To avoid this source of inaccuracy it is best to employ, as the testing liquor, a saturated solution of ammonium sulphate, which, being very soluble both in hot and cold water, is not precipitated. One part of the ammoniacal salt is equivalent to 1·32 parts of potassium sulphate, or 1·13 of potassium chloride.

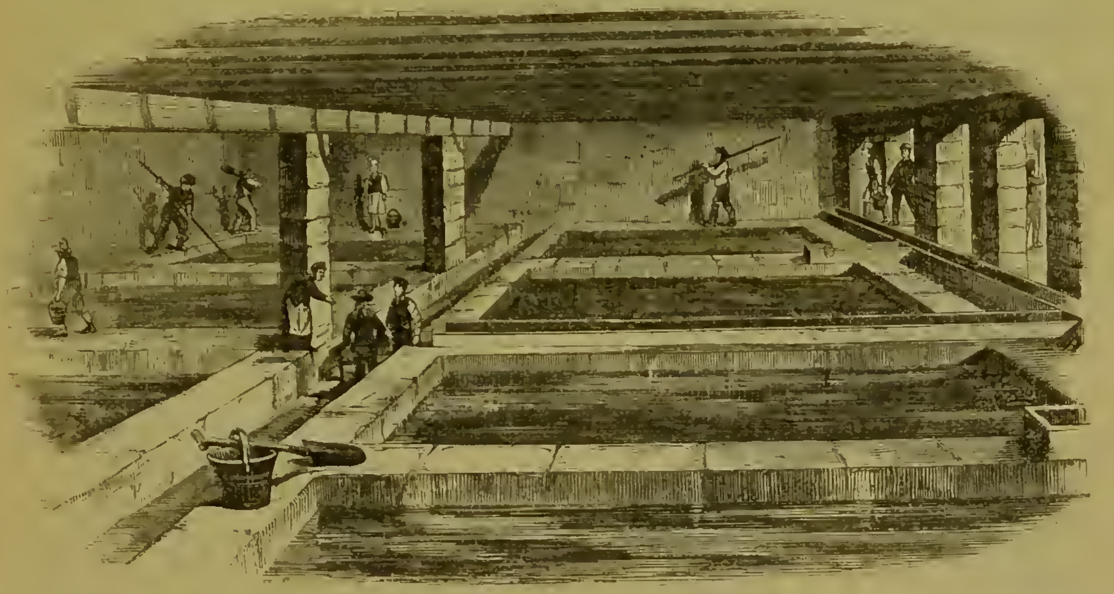
Potassium chloride for alum making is obtained in large quantities from the waste liquor of the soap works, from saltpetre refineries, and from the glass-houses. Wood ashes should never be used, on account of the lime which they invariably contain.

The ammoniacal reagent is obtained from the liquor from gas works; this is converted into ammonium sulphate either directly by the addition of sulphuric acid, or by treatment with sulphate of lime or iron. The crude ammoniacal liquor obtained from the manufacture of ammonium chloride, by the destructive distillation of animal matters, may be employed with equal advantage for the same purpose.

Having prepared the aluminous liquor, and ascertained the amount of salts contained per gallon, the crystalline flour is precipitated by gradually adding to the aluminous liquor the concentrated solution of the precipitant, the mixture being kept briskly agitated till the whole of the powder is thrown down. A slanting plane is fixed adjacent to the precipitating vessels, on which the workmen shovel the subsided powder, for the purpose of freeing it from the impure mother liquor of the vessel; the portion of lie adhering to the crystals drains off, and runs back to the mother liquor; still it does not wholly separate by this means, a small portion being mechanically retained by the mass of powder, which produces the brownish-yellow colour of the crystals after the first draining. To remove the colour recourse is had to another process called "washing." For this, it is sufficient to stir the powder with a small quantity of water in an appropriate tank or vat. The impurity mixed with the crystals is taken up by the water, as likewise a small portion of the alum, and when the substance has subsided the liquor is drawn off by means of a siphon; two washings are found sufficient to separate all adhering foreign soluble bodies. The water employed in washing the flour should be of as low a temperature as possible, as the quantity of alum dissolved out is in proportion to the temperature of the water.

Fig. 5 is a view of the precipitating and crystallizing cisterns at one of the Hurlet works; they are constructed of stone, well adjusted and embedded in tempered clay, on a site contiguous to the evaporating furnaces. Having ascertained the quantity of sulphate of alumina per gallon in the concentrated liquor in those boilers, their whole bulk of lie is found by gauging, at the temperature of 60° Fahr. (15°·5 C.). A corresponding amount of potassium chloride, or ammonium sulphate, is then weighed out and placed in one of the cisterns, after which the plug is removed from the boiler, and the liquor conducted along stoneware gutters to the coolers till it flows upon the heap of chloride. Sometimes the latter is gradually added to the liquor as it flows along in the gutters. Towards the end the mixture is well agitated to insure a complete solution of the alkaline salt and a uniform state of the liquor. In four or five days, when the plug is drawn out from the cistern and the mother liquor run off, the bottom and sides of the cooler are found lined with crystals of alum, generally some inches in thickness. This is called *first alum*, and is removed from the vessel to the draining stage, which is a raised platform in a sloping position, so that the mother liquor drains off from the heap, as shown in Fig. 6.

Fig. 5.



The first alum, when drained, is washed to remove as much of the mother liquor from it as possible. For this purpose tubs are filled with second alum mothers, or liquor which has been used in washing in a subsequent part of the process; and the alum, placed in wooden sieves, is rinsed in this liquor, and thrown on the stage.

The second alum boiler, which is a deep stone cistern covered with wood, Fig. 7, is then prepared by running in a quantity of liquor which had been previously used in washing the second alum; steam is next introduced, till the liquor has nearly attained its boiling point, when the washed and drained crystals are thrown in, and constant agitation kept up. This is continued till as much alum has been added as will increase the specific gravity to 60° Twaddle, and the temperature is raised as much as possible by the steam, which is then shut off, and the boiler being closely covered up, the liquor is allowed to remain at rest for twelve hours, for all the impurities to settle; the plug is then removed, and the clear liquor runs off to the second alum coolers, which, like the preceding, are sunk stone cisterns. In these it remains for four or five days, when the sides and bottom are found lined with alum, now nearly white, to the thickness of 8 or 9 inches. The small cup-like clusters of second alum crystals, which form on the surface of the coolers at this stage of the process, and successively fall to the bottom, assume the beautiful appearance exhibited in Fig. 8. The mother lie, which should have a specific gravity of 26°, is removed, and used for washing up the first alum as already described; and the crystals being broken up, are rinsed with pure water and drained, previous to the finishing process.

Fig. 6.

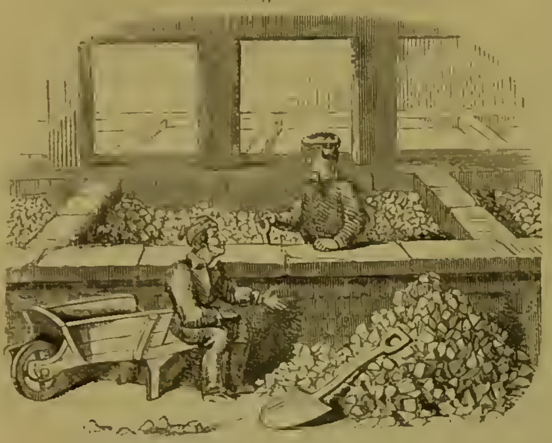
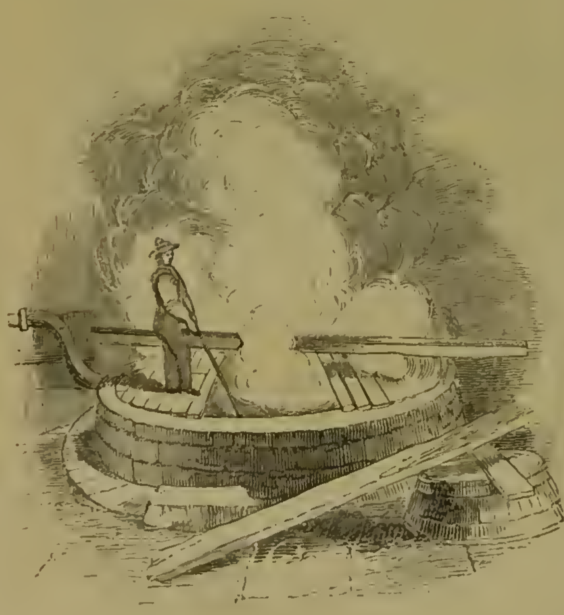


Fig. 7.

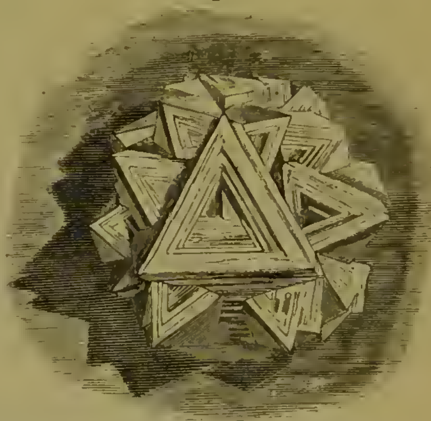


Crystallization.—The powder or alum flour, consisting of minute or imperfect crystals, prepared as just mentioned, is very seldom sent into the market, the consumers preferring to use the salt in the form of large crystals, the alum being less liable to be adulterated with any other impurities when in a well-defined crystalline state than when it is amorphous.

Crystallization of the alum flour is effected by dissolving it in as little boiling water as possible, and when no more of the substance is taken up, running the saturated solution into crystallizing vessels, which are denominated "roching casks," or "growing vessels." Steam is better adapted than water for the purpose of dissolving the flour. A large wooden vessel, lined with sheet-lead, is provided for this purpose, and in the interior the powder is sustained occasionally on perforated shelves, so that the steam which issues from the pipe that enters at the bottom may have more extensive contact with it, and thus dissolve it more readily. As the steam penetrates through the several diaphragms bearing the powder, part is dissolved; that which is liquefied in the upper ones is concentrated by the hot steam as it falls down to the bottom of the vessel, and being hot, it is at once run into the vessels for crystallization.

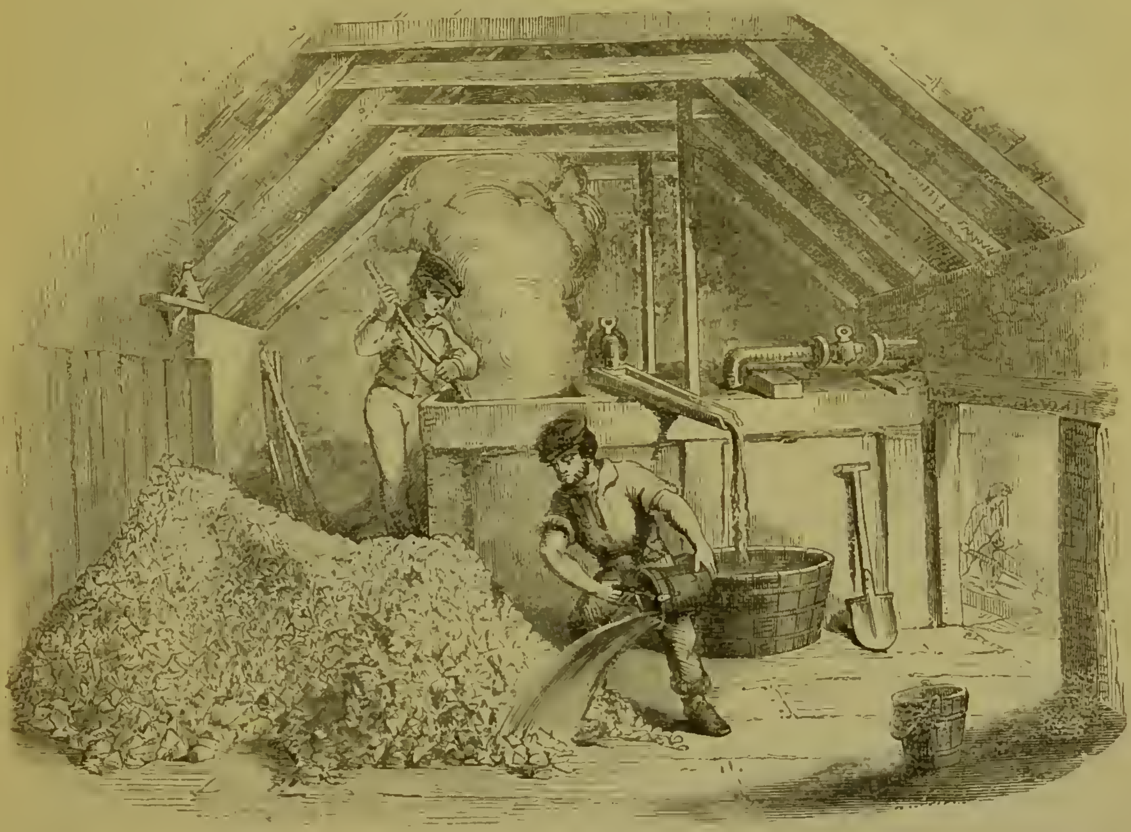
Fig. 9 is a drawing representing the roching-pan as it is usually found in the alum factories of England and Scotland; it works on the above principle, and therefore renders a subsequent evaporation unnecessary. It is a large covered leaden cistern, into which steam is forced, and the alum is shovelled in to meet it; a solution takes place rapidly, without any addition of water. The temperature of this solution having reached 96° Fahr. (35°·5 C.), the heat is raised as high as possible, generally to 224° Fahr. (106°·6 C.); and the steam being shut off, the pan is closely covered up, and allowed to remain at rest for four or five hours, or till the temperature has fallen to about 200° Fahr. (93°·3 C.) The water which has been used for washing the crystals, is taken again for washing the first alum, as above.

Fig. 8.



At Valmunster this operation is carried on in vessels constructed as follows:—The wooden troughs are lined with lead in the ordinary way, but are closed by a lid in which are two openings; one of the openings receives the steam-pipe, and the other a wide leaden funnel. The sides of the funnel are perforated with small holes, 300 of which are inserted in every square foot of surface. In this funnel the alum flour is placed, and the steam injected through the pipe, in seeking to escape through the apertures in the funnel, dissolves the powder; the solution trickles to the bottom of the vessel at a temperature of 212° Fahr. (100° C.). At the termination of the process a small quantity of undissolved basic alum is left in the funnel, which is added to the concentrated liquor ready for precipitation, and is redissolved as before stated. During the time the flour is dissolving in the roching vessel, the crystallizers are being prepared, and the aluminous solution, of a density about 1·485 to 1·515, is run into them by appropriate means.

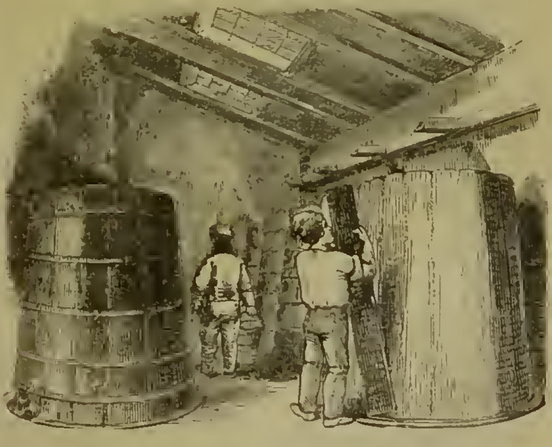
Fig. 9.



The crystallizers are large wooden casks, 5 to 8 feet high, 3 or 4 feet in diameter at the top, and tapering in a somewhat conical form towards the bottom. The staves of these casks are very strong, nicely adjusted, lined with sheet-lead, and held together by means of strong iron hoops, which are hammered on when required for use, and may be as easily removed.

Fig. 10 is a view of these casks arranged in the crystallizing shed; over each a spout is fixed to fill them with the liquor. Some of the casks are represented as on the point of being charged, while in others the crystallization is completed, and the staves are being taken off. When the casks are charged, they are covered up, each with its own lid. After remaining undisturbed for four or five days, according to the state of the weather, the hoops are loosened and the staves removed; the crust of alum then formed being sufficiently strong to resist the pressure of the interior liquor. In this state they are left for a fortnight, in order to allow the whole of the crystallizable salt to form. The workmen then pierce a few holes in the sides of the column with an axe, and the mother liquor in the interior flows into the gutters in the stone floor, which conduct it into an appropriate cistern, whence it is pumped up to dissolve fresh quantities of the pre-

Fig. 10.



pared alum flour. When the roching casks are dismantled, and the alum of the upper part removed, there is invariably found at the bottom a considerable quantity of white slime mixed with small octahedral crystals of alum. A quantity of this slime, well washed, gave the following centesimal composition:—

	Per Cent.
Sulphuric acid,	39·40
Alumina,	31·80
Potassa,	10·03
Moisture,	16·70
Insoluble matter,	2·07
	100·00

On examining the insoluble portion, it was found to contain—

	Per Cent.
Silica,	73·79
Alumina, with a trace of iron,	21·83
Lime,	3·49
Loss,	·89
	100·00

The mother liquor which remains after the separation of the alum, is composed of solutions of ferrous and ferric sulphates and chlorides, and magnesium and other alkaline sulphates; and when soap-boiler's waste has been used as the precipitating agent, it contains, in addition to the foregoing, sodium alum in moderate quantities; if bisulphate of potash, saturated with wood ashes, has been employed, the liquor will contain some sulphuric acid and other ingredients in small proportions. From a chemical analysis of the alum shale, or ore, in the first instance, a good idea can be formed of the principal bodies remaining in the mother liquor, and their utility for the formation of secondary products; for instance, if free acid should be detected in the lie, it may be employed to neutralize crude ammonium carbonate from the gas-works, to procure ammonium sulphate; or if much iron be present in the acid liquor, a further addition of borings of iron, or old iron, may be added, and in this way ferrous sulphate for manufacturing purposes formed. Should the mother liquor contain large quantities of the mixed chlorides of iron, it may be evaporated to dryness, and the dry mass heated to redness in a reverberatory furnace to drive off the hydrochloric acid, and thus ferric oxide obtained of a quality fit for use as a pigment.

Magnesium sulphate, Epsom salts, is also manufactured from the mother liquors of the ores.

The mass of alum from the crystallizing tub is cut into square blocks for market, when it will be found interiorly to consist of beautiful large octahedral crystals, similar to Fig. 11, which project from the sides and cover of the vessel.

For some purposes the alum is preferred in the form of a fine powder or flour; a portion is therefore prepared for the market in this state. Fig. 12 is a sketch of the machinery usually employed at the alum-works for this purpose. The alum, reduced to coarse fragments, is crushed beneath a pair of ponderous wheels or rollers of cast iron, one of which is rough or fluted on the rim, and the other perfectly smooth. These traverse in a circle, and the alum is constantly pushed into the path of their revolution by two vertical revolving blades, one of which sweeps it inward, and the other outward. The grooved roller crushes the larger fragments before they are subjected to the action of the smooth surface of the other. When one charge is thus reduced to powder, it is shovelled out by an attendant to make room for a new supply; and the powder is passed by hand into a hollow cylindrical sieve, shown at the right hand of the figure. The lower portion, or belly of the cylinder, which lies in a slightly inclined position, consists of fine wire gauze; the upper convexity is made of white iron. This is surrounded by wooden hoops, to which are attached brushes that pass into the interior of the cylinder. An oscillating motion is given to the latter, and that portion of the alum powder which has been reduced to a state of sufficient fineness is driven by the action of the brushes through the sieve, from which it falls in a beautiful white flour into a box below; the coarser particles passing onward to the other end of the cylinder, to be again subjected to the action of the crushing rollers.

Fig. 11.

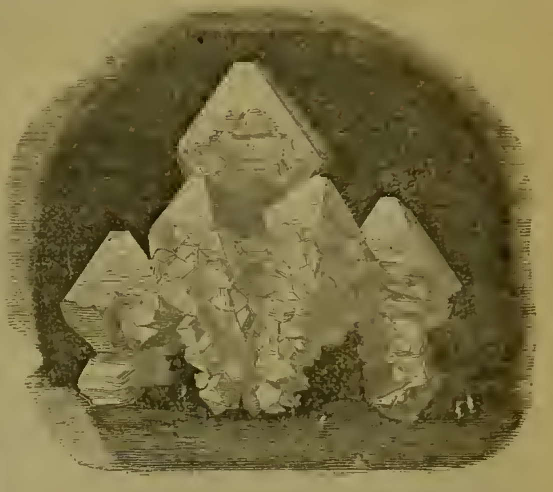
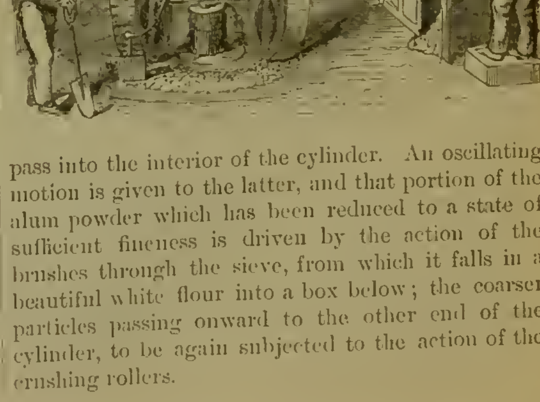


Fig. 12



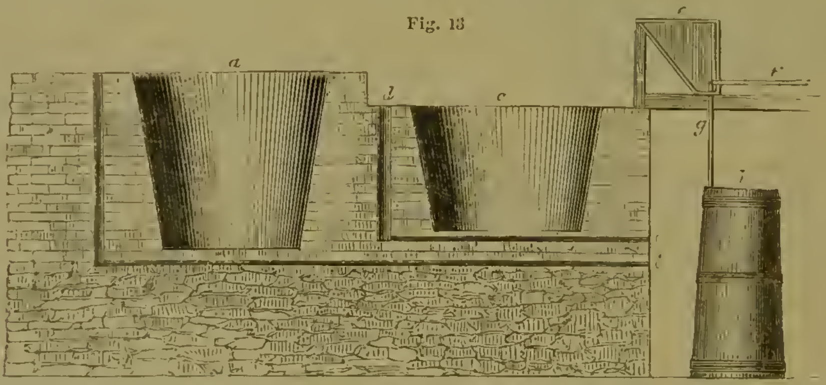
The most important changes which the manufacture of alum has undergone in the last ten years are the substitution of ordinary sulphuric acid for the sulphuric acid generated by the prolonged exposure to the air of iron pyrites, and the general substitution of ammonia for potash. The direct treatment of aluminiferous minerals with sulphuric acid was patented in 1845 by P. Spence, of Manchester, and was brought into practical use in 1847 by the firm of Spence & Dickson. The importance of this step may be judged from the fact that while formerly 60 tons of the Berkshire oolitic schist were required to produce 1 ton of potash alum, the manufacture of 1 ton of ammonia alum by the new process does not take more than three-quarters of a ton of the same mineral. The schists used by Mr. Spence are found under the coal-beds of South Lancashire. This schist, which derives its black colour from the presence of carbonaceous matter, is piled up in heaps of from 4 to 5 feet in height, and calcined, as already described, at a heat bordering upon redness. The object of this process is to make the alumina more readily soluble in sulphuric acid. If the temperature is too high, a partial vitrification (fritting) results, and the alumina is rendered almost insoluble in the acid. The calcination lasts ten days, a certain quantity of fresh schist being added daily. When the operation has been well conducted, the residue is soft and porous, and of a light brick red. It is placed in covered vessels, each capable of holding 20 tons, and digested for thirty-six to forty-eight hours with sulphuric acid of spec. grav. 1·35. The mixture is kept at the heat of 230° Fahr. (110° C.), by a fire beneath, and by driving in the vapour of an adjoining boiler containing gas-liquor. This simultaneous treatment gives excellent results, provided that the acid is always kept in excess. The ammoniacal salts pass into the digesters, and are there decomposed by the acid. The excess of ammonia is afterwards set at liberty by the addition of a certain quantity of lime. The ammoniacal liquor flows into cisterns, and is stirred continually while cooling, in order to determine the formation of small crystals. These crystals are subsequently dried, and then washed with the mother liquor that drops from the blocks of alum. Not a trace of iron is found in these crystals, though the mother liquor contains it in abundance in the state of sulphate. This result is commonly obtained by means of rapidly redissolving and recrystallizing the alum. Spence arrives at the same result by means of steam, without addition of water as a solvent. The crystals are thrown into a hopper, at the bottom of which they come in contact with a current of steam, which rapidly dissolves them. The crystals and the steam are in such proportions that all the crystals are dissolved when all the steam is condensed: 4 tons of crystals are thus dissolved in thirty to forty-five minutes. The solution flows into a leaden tank, where it stands for three hours, depositing a certain quantity of matter insoluble both in acid and water—probably basic sulphate of alumina. The liquor is thence run into cylinders, the bottoms of which are made of Berkshire flagstone, and which are 2 yards in diameter, with movable sides. After standing from five to eight days, the sides are removed, and a cylindrical mass of crystallized alum is found. After standing again for eight days, a hole in the bottom of the cylinder is opened, and a quantity of liquid escapes. The mass is generally 18 inches thick at the bottom, and 1 foot in the sides, and contains 3 tons of marketable alum. The liquid which escapes contains another ton. The blocks of alum obtained by this process often present a faint rose shade, which a hasty observer might attribute to metallic impurities. This tint is due to the presence of organic matter, probably a trace of aniline, derived from the gas-liquor.

The substitution of ammonia for potash is not peculiar to those establishments which make direct use of sulphuric acid. In consequence of the rise in the price of potassic salts, ammonia has been introduced into the manufactories which still work on the old method, not only in England and in France, but in Germany. In 1851 the great Bonner Works made exclusively potash alum. Now, with the exception of some 300 tons, its entire yearly production is ammonia alum. Many samples sold as potash alum contain a proportion of ammonia.

Production of Alum from Rocks containing Alumina.—Alum has been produced from various rocks of which alumina forms a constituent, by a process similar to that which naturally gives rise to this body in volcanic districts. The operation is confined to one or two isolated works on the Continent, and those are conducted in juxtaposition with metallurgic operations where sulphurous acid is in abundance. At Lintz, on the Rhine, where there is a spelter work and plenty of zincblende, zinc sulphide, the sulphurous acid evolved during the roasting of the ore is conducted over the aluminous mineral moistened with water. Basalt and copper slate are the materials employed, and when these are exposed to the action of the gas and sufficient moisture and air, decomposition ensues, aluminium sulphate, with sulphates of iron and copper, are formed, which are separated from the insoluble residue by steeping with water, and the subsequent treatment for obtaining the alum is on the same principle as those methods which have been already described. In such operations there is always a quantity of sodium alum produced, on account of the presence of this alkali in the basaltic mineral. The sodium alum is formed from the commencement, and the amount corresponds of course with that of the soda in the mineral, which in many basalt rocks is from 5 to 6 per cent.

Alum from Clay.—Chaptal and Alban, in France, devised a different process for the preparation of alum from clay. Clay consists of basic aluminium silicate, free alumina, and by the action of sulphuric acid aluminium sulphate is formed with separation of silicic acid. Pipe-clay answers the purpose best; but that the acid may more readily penetrate the mass, the clay is submitted to a previous calcination to increase its porosity, and more fully oxidize the iron which it may contain. Fresh clay, when employed, requires a considerably longer time than when it is calcined and pulverized previously to the addition of the acid; if it be found requisite, the ground material is sifted: 45 per cent. of the weight of the clay of sulphuric acid, of 45° B., is poured upon it in a cistern which is heated, by the surplus heat from the calcining furnace, to about 158° Fahr. (70° C.). Decomposition ensues, and the mass assumes greater consistence from the liberation of silicic acid; in this state the contents of the cistern are removed to the open air, where they are allowed to remain for a space of some months, to allow of the complete disintegration of the clay by the acid. The mass is afterwards lixiviated and the aluminium sulphate treated as in ordinary cases. Clay with as little iron and lime as possible should be selected. Some manufacturers employ large shallow troughs, lined with lead and heated from below by flues, for the purpose of mixing the prepared clay with sulphuric acid, in which it is frequently turned over until the acid has combined with its alumina. In this part of the operation an improvement has been lately effected, which consists of distributing the clay upon a shallow circular trough, and permitting the proper quantity of acid to flow in several small jets from a cistern placed above, whilst an agitator, worked by machinery, mixes the the clay and acid thoroughly. In this way a speedy combination is effected. After the termination of the action of the acid the contents of the cistern are removed to the stone vat, *a*, Fig. 13, where they are treated with successive quantities of water, to extract the aluminium sulphate. Each lie is siphoned off into the pipes, *b b*, which convey it to the evaporating pans, to be boiled down to the proper strength for precipitating with the alkaline salt. When this point is attained the liquor is pumped up into the cistern, *c*, which is similar to *a*, already described, and called the precipitating vat; here the appropriate quantity of potassium sulphate is added. On agitating the solution the "alum powder" precipitates; this is allowed to subside, and the mother liquor is drawn off to the pipes, *d d*, by a syphon. If the mother liquor be not very impure it is employed for a subsequent precipitation. The precipitated alum is washed in the cistern, *c*, with successive small portions of water, each liquor being drawn off repeatedly in the same way as the mother liquor.

Fig. 13



When the powder is purified by repeated washings, it is ladled out into the funnel-shaped vessel, *e*, where it is very economically dissolved, by injecting steam upon it through the pipe, *f*, and the saturated solution of alum descends to the crystallizing frame, *h*, through the pipe, *g*.

The evaporation of the various liquors is conducted in boilers lined with lead, by means of convoluted steam pipes placed in the liquid. This plan of concentration of the liquid is found to be best adapted for such liquors, as well as being cheaper.

Taylor, of Bristol, manufactured alum from pipe-clay by calcining it in the usual way, and then treating it with its own weight of sulphuric acid, of specific gravity 1·200, in a large tub, upon the bottom of which a steam-pipe is coiled for the purpose of heating the mixture to about 200° or 212° Fahr. (93°·3 to 100° C.); furnace clinkers and pieces of pottery, or other matters, are also strewed upon it to facilitate the filtration of the solution of aluminium sulphate. After the mixture of clay and acid has been heated for twelve hours, the solution is drawn off through a pipe at the bottom of the tube to the precipitating vats at a density of 1·300.

Pochin, of Manchester, first prepared alum cake from china clay. This product is much esteemed on account of the large amount of aluminium sulphate it contains. A clay is chosen which is as free from iron as possible, and heated gently in a furnace with access of air, in order to remove the water and render the clay more soluble in acids. By losing its water the clay becomes very porous, and takes up the acid with great avidity by capillarity. The gently ignited and powdered clay is then gradually added to sulphuric acid, of specific gravity 1·52, in leaden pans: here the mass thickens, heats violently, and boils; it is then transferred to iron tanks, where it ultimately solidifies. The porous mass is perfectly dry, though still retaining a large amount of combined water. It also contains all the silica originally in the clay, but in an extremely fine state of division. If it is desired to convert the alum cake into alum, it is lixiviated with the washings from alum flour; and the solution, after standing to become clear, treated with potassium bisulphate, or ammonium sulphate from gas liquor, in the usual way.

From Felspar.—This mineral, which is a double silicate of alumina and potassa — $AlKSi_3O_8$, or

$AlKSiO_4,2SiO_2$—is decomposed by potassium sulphate in a reverberatory furnace, and then fused with carbonate of potash to a glass; on treating this with boiling water decomposition follows, a soluble silicate of potash dissolves, while an insoluble double silicate remains. The insoluble portion, when treated with boiling sulphuric acid, is decomposed into alum, and silica separates. The alum is dissolved out by water, and the solution evaporated for crystallization; the soluble alkaline silicate may be turned to account by combining the silica with lime.

Turner was the first to carry out the proposition of Sprengel, of converting felspar into alum. A patent was granted to him in 1842 for his process, which he describes in his specification as follows:—If desirous of making potash alum, the best substance to operate upon is a potash felspar. This felspar is ground in a common edgestone mill, till the powder is like fine sand, a process which is much assisted by heating it to redness, and then plunging it in cold water; it is afterwards mixed with its own weight of sulphate of potash, and placed in the upper part of the inclined bed of a reverberatory furnace, similar to the annexed Fig. 14—known in the potteries as a *frit-furnace*—previously brought to a red heat. When the glass produced by the fusion of the powdered mineral and the alkaline sulphate has been observed to flow down to the bed of the furnace, there is added gradually, at the lower end of the furnace, as much carbonate of potash as was previously used of sulphate. This operation of adding proportionate quantities of carbonate of potash to the molten mass as it flows down into the lower part of the furnace, is continued till the "sack" of the furnace is quite full, the mineral and sulphate being introduced at the upper end: the glass is then fit for the next operation.

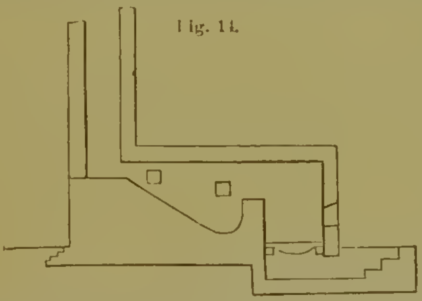


Fig. 14.

The compound might be prepared in a furnace with a flat bottom, from which it can be separated with greater facility; in this case, however, no carbonate should be used till the whole of the sulphate has been seen to be decomposed, after which it is to be introduced, as in the preceding instance, and fused with the mass. On boiling the glass produced in this manner with water, the same quantity of potash as was added to the felspar, and two-thirds of the silica contained in the mineral, are dissolved, while the remaining one-third of silica, and the alumina, and an equal quantity of potash as the felspar originally contained, are left in the form of a light porous substance, which is carefully separated from the solution, and washed well with water to withdraw potassium silicate; next, the porous precipitate is put into a large leaden cistern or boiler, and acted upon with boiling sulphuric acid, of specific gravity 1·20, which will contain sufficient water for the solution and crystallization of the alum formed by the decomposition of the mass. As a general rule, as much acid should be added in the diluted state as will contain 160 lbs. of dry acid to every 285 lbs. of felspar employed. The boiling liquor, after the sediment has subsided, is drawn into coolers, such as are generally used for crystallizing alum; here about four-fifths of the alum contained in the liquor will separate into crystals. From this mother liquor the residual alum is obtained by evaporating it to dryness; by this means the silica present in the solution is rendered insoluble, and the dry mass is acted upon either with water or a further quantity of mother liquor, and the solution evaporated to the crystallizing point as before. When ammonium or sodium salts are used in the above process, ammonium and sodium alums are produced.

With the exception of the high temperature required to decompose the felspar, Turner's process is very successful, but this was found to be a serious drawback when working on the large scale.

Alum may likewise be obtained by treating a mixture of two parts of felspar and three of fluorspar with sulphuric acid at a low red heat, until vapours of hydro-fluosilicic acid cease to be evolved. The decomposition of the fluorspar by the sulphuric acid causes the liberation of hydrofluoric acid, and this in turn decomposes the silicates, combining with their silicic acid to form the evolved hydro-fluosilicic acid. Sulphate of lime is formed in the first stage of the process; but this is subsequently decomposed, and potassium and aluminium sulphates formed.

The mass is lixiviated with water, and after the separation of some sulphate of lime, the solution is evaporated, and on cooling it deposits an abundant crop of nearly pure alum crystals.

From Cryolite or Greenland Spar.—This mineral has the composition:—

Aluminium,	13·0
Fluorine,	54·5
Sodium,	32·5
	100·0

and is represented by the formula, $Al_2Na_3F_6$. It has of late years been much used for the production of soda and alum. Its decomposition is effected in the dry way by igniting it with calcium carbonate in the proportion of 6 equivalents of Ca_2CO_3 to 1 of cryolite, when

$$\overset{\text{Cyrolite.}}{Al_2Na_3F_6} + 6Ca_2CO_3 = \overset{\text{Calcium Fluoride.}}{6CaF} + \overset{\text{Sodium Aluminate.}}{Al_2Na_3O_3}$$

The sodium aluminate is extracted with water decanted from the heavy calcium fluoride. Carbonic acid gas is then passed into the solution, when sodium carbonate is formed, and gelatinous alumina is precipitated. This precipitate is collected, dissolved in dilute sulphuric acid, and evaporated to obtain aluminium sulphate, or treated with an ammonium or potassium salt to convert it into alum:

100 parts by weight of cryolite yield 305 parts of alum.

Cryolite is also decomposed by boiling it, after having pulverized it very finely, with lime and water in a leaden pan. The result of the interchange of constituents is the same as that given above, aluminate of soda and fluoride of calcium being produced. When the heavy calcium salt has settled down the clear liquor is decanted and the sediments washed, the first washings being added to the original liquor, and the second and third washings used instead of pure water at a subsequent stage. The alumina is then precipitated by adding finely pulverized cryolite in excess, thus:—

$$\underset{\text{Sodium Aluminate.}}{Al_2Na_3O_3} + \underset{\text{Cryolite.}}{Al_2Na_3F_6} = \underset{\text{Alumina.}}{2Al_2Na_3F_6} + \underset{\text{Sodium Fluoride.}}{6NaF.}$$

The gelatinous alumina is allowed to settle, and treated by any of the methods previously described.

SPENCE of Manchester has recently patented a process for the manufacture of alum from Rodonda phosphates, a mineral from the West Indies containing alumina with phosphate of iron in variable quantity.

The treatment for the aforesaid purposes of the said minerals may be varied in details, but the following is a description:—The raw mineral is calcined in kilns (similar to those used for lime) by mixing it with coal or coke and exposing it to a red heat, or else it is ground sufficiently fine to pass through a sieve of twenty meshes to the inch. The former plan is, however, preferable, as it facilitates the solution of the mineral substance, and renders a portion of the iron insoluble by oxidation. The mineral having been prepared by these or similar means, is then placed in leaden vessels, and an equal weight of sulphuric acid of spec. grav. 1·6 added to it, if it contains 20 per cent. of alumina, but only three-fifths of its weight if it contain 12 per cent., and in similar proportions for other degrees of richness. Heat is then applied by blowing steam into the vessel containing the mixture. The mineral dissolves and the specific gravity rises. This is cautiously reduced by addition of water or weak liquors from subsequent parts of the process, the whole being kept constantly boiling until all is dissolved except the insoluble sediment, and the strength of the liquor becomes 90 Twaddle, or 1·45 spec. grav. This liquor is next passed into a close leaden vessel, and vapour distilled into it containing ammonia obtained from gas ammoniacal liquor, subjected to boiling either by fire or steam injected into it. The quantity of gas liquor I used being from 600 to 900 gallons to each ton of the mineral, according to its richness. When all the ammonia has been distilled into the mineral liquor it is allowed to settle for a few hours, and all the clear solution (now at a strength or spec. grav. of 1·4 or 80 Twaddle) is run off into lead coolers, where the alum crystallizes out in the usual way. The liquor remains in these coolers for some days, with frequent stirring, in order to obtain all the alum possible. SPENCE finds that when the mineral contains 20 per cent. of alumina, that he obtains about 1½ tons of alum from 1 ton of the ore.

The mother liquors having deposited all the alum that can be obtained, is now chiefly a solution of phosphoric acid with a small quantity of aluminium and iron sulphate, and ammonium sulphate or phosphate of ammonia. This liquid may be used directly as a fertilizing agent, but is usually mixed with dry sawdust or other absorbing agent, in just sufficient quantity to absorb all the liquor. It is then dried at a low heat, so as not to char the sawdust; and when dry it forms an artificial manure containing phosphoric acid and ammonia, in such quantities and condition as to make it a valuable fertilizer. Instead of ammonia gas liquor used with the mineral solution to produce alum, salts of potash may be used either alone or in combination with ammonia; of the former, the chloride of potassium of commerce, or preferably sulphate of potash, as although chloride of potassium will yield a sufficient product of alum, the fertilizer would from its use have a tendency to deliquesce, but sulphate of potash will not have that effect.

WILSON, of Glasgow, patented a process for the manufacture of alum, the chief novelty of which consisted in heating the liquids for the digesting the shale, and in applying the mother liquors after precipitating or separating the alum, so as to make the sulphuric acid, either free or in combination with other bodies, available in subsequent operations.

The mixture of sulphuric acid and water is warmed in a separate vessel, and then run into the digesting tank containing the calcined shale, at a temperature between 150° and 200° Fahr. (65°·5 to 93°·3 C.), and the heat maintained at the proper degree by conducting the waste heat from the furnace of the heating vessel below, through flues under and around the digesters.

To prevent the loss of sulphuric acid, the ordinary method is varied in the following manner:—The strong mother liquors, left over after depositing the alum formed from the combination of the aluminium sulphate with the ammonium sulphate, are not used again to dilute the acid, but are employed with fresh ammoniacal liquor from the gas works or elsewhere, to form sulphate of ammonia. The decomposition of the portion of alum which the mother liquor held in solution is guarded against by adding the ammoniacal liquor in such quantities, that a slight portion of the acid in the liquor is uncombined. A few trials enable the operator to judge, by means of test papers, the proper point at which the neutralizing should be suspended.

When the solution of the mixture of ammonium sulphate and alum, after filtration or reposing to allow impurities to deposit, is sufficiently concentrated by evaporation, it is run in among the solution of aluminum sulphate from the digesters, and the alum which it contains is deposited with that of the fresh liquor.

RICHARDSON, in 1850, sealed a patent for further improvements in the manufacture of alum, the leading parts of which are as follow:—In lixiviating the calcined shale for the purpose of obtaining the raw alum liquor, a series of pits, such as is seen in the annexed figure, is employed. Fig. 15 shows the manner of effecting the solution of the aluminium sulphate. B^1 B^2 B^3, &c., are a series of pits, furnished with pipes, A^1 A^2 A^3, &c., for the purpose of conveying mother liquor from one to another, the pipes being stopped with taps or cocks, as seen in the figure. Water is run from the service tank, D, through the pipe, C, into the pit, B^6, till it flows over through the pipe, A^6, and passes through the pipe, E E, till it enters B^1, through which it rises till the pit is filled, exhausting the contents of the soluble ingredients in its ascent. When the liquor has stood for some days, a fresh supply of water is continued as before in B^6, by which the liquor in B^1 is forced over through A^1, the plug being removed into the pit, B^2, containing fresh calcined shale; the liquor ascends through the shale in this pit as in B^1, and when it has remained two or three days in contact with it, a fresh supply of water is allowed to flow into B^6, which forces the liquid contents of B^2 into the third tank, and the treatment repeated as long as the workmen find it necessary to bring the liquor to 28° Twaddle, or such higher strength as is required; and when the proper degree of saturation is attained, the solution is drawn off by the stopcock, F. As soon as the shale in any of the pits is found to be exhausted, it is removed, and a fresh supply introduced, and the lixiviation proceeded with as before.

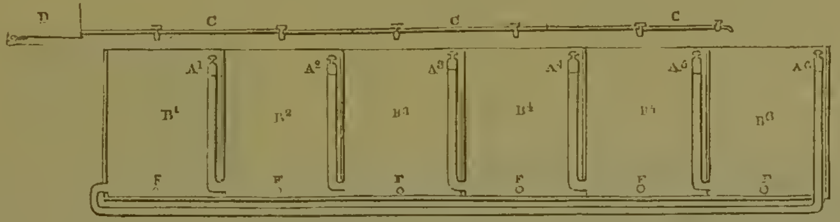
Of course, as the operation goes on week after week, it will become necessary to commence the supply of water from the service tank, D, to the different pits in succession, which may be done as above described, or by allowing it to run at once down the pipes, A A A, from the respective taps in the pipe, C. By these arrangements the labour of pumping the liquor from one pit to the other will be saved, the process generally rendered more convenient, and the result will be a more perfect exhaustion of the shale.

PRODUCE.—The great variation in the character and richness of the ores does not admit of even a proximate relation being established between the quantities employed and the amounts of alum produced in different works. At Valmunster, from 17,300,000 lbs. weight of alum shale, 400,000 lbs. of alum, or 2·3 per cent., are obtained, for the production of which 100,000 lbs. of sulphate of potassa are required, and 1,400,000 lbs. of coal consumed.

The slate at Liége yields 2 per. cent of alum, and that from other localities only from 1 to ½ per cent.

IMPURITIES.—Alum always contains a little iron; that from Liége contains 0·02 per cent.; that from Gavelle, near Paris, 0·08 per cent.; that from Aveyron, 0·11 per cent.; and English alum about 0·12 per cent. This amount of iron is sufficient to impair the delicate colours used in calico-printing; it can, however, be removed by recrystallization, or as prussian blue, by means of potassium ferrocyanide (K_4FeCy_6). The Roman alums—a name given to all varieties of alum imported from Italy—are sufficiently pure for immediate use, and are highly prized on account of the minuteness of the quantity of iron which they contain.

Fig 15.



Roman alum commonly consists of small disconnected crystals, covered externally with a kind of reddish mud; on the alum being dissolved in water none of this coating is acted upon, and the quantity of iron alum present in the solution does not exceed 0·005 per cent. Alum of this description produces a blue precipitate with potassium ferrocyanide only after standing for several hours, while ordinary alum affords a precipitate in a few minutes. Roman alum is generally crystallized in the form of octahedrons, though cubical crystals are occasionally found. Its reactions differ from those of ordinary alum in many respects.

CHEMISTRY OF ALUM.—Potassium alum requires about 18 parts by weight of cold, and less than 1 part of boiling water to dissolve it, and crystallizes very readily in large regular octahedrons, of which the apices are always more or less cut off. Its taste is sweetish, and very astringent. It is an acid salt, reddens vegetable blues, and dissolves metals with evolution of hydrogen; in the air it changes very slightly. Its water of crystallization amounts to 45·5 per cent. of its weight, or 12 atoms; thus it loses its water of crystallization with great facility at 197°·6 Fahr. (92° C.). The fused salt in losing this water becomes viscid and intumesces, leaving calcined or burnt alum, which is sometimes used as a caustic which dissolves in water, although but slowly.

The octahedral crystals of potassium alum consist of sulphuric acid, aluminium, potassium, and water. and have the formula $AlK(SO_4)_2 12H_2O$.

When octahedral alum is digested with hydrated alumina, and the solution allowed to evaporate spontaneously by exposure, cubical crystals are formed with a simultaneous separation of a basic salt of alumina; the crystals have an acid reaction, and the same properties as ordinary octahedral alum.

LOEWEL prepared pure potassium and ammonium alums, crystallized under the ordinary octahedral system, and obtained cubic crystals of potassium alum by dissolving the octahedral crystals in three or four times their weight of water, heated from 72° to 81°

Fahr. (22·°2 to 27°·2 C.), and adding an aqueous solution of potassium hydrate in alcohol in successive small portions, till the subsalt of alumina, thrown down by each addition, was no longer dissolved upon the agitation of the liquid. The form assumed by the salt after adding a quantity of potassium equal to about one and a half equivalents aluminium (that is, on taking from the aluminium sulphate contained in the alum nearly half its acid), was that of truncated and cubical octahedrons; but on the further addition of alkali, cubes were formed on spontaneous evaporation of the liquid.

Cubical ammonium alum he procured by dissolving the pure octahedral salt in three to four times its weight of water at 113° Fahr. (45° C.), and adding ammonia till the small quantity of subsalt ceased to dissolve in the solution.

When some of the solutions thus prepared had given a certain quantity of cubic crystals the solution became cloudy, and deposited a gelatinous tribasic aluminium sulphate ($Al_2O_2(SO_4) + 9H_2O$).

The potassium and ammonium cubic alums resist the action of air dried by sulphuric acid in a bell-jar for weeks without efflorescing. LOEWEL suggests that it would be more correct to state that no efflorescence takes place. He placed crystals of pure alum under bell-jars, in which the air was dried by sulphuric acid; at first the crystals lost some thousandths of their weight of interposed water, but afterwards remained for several months, even for a whole year, at a temperature rising to 77° Fahr. (25°·0 C.).

According to DE HAUER a crystal of cubic alum when placed in a solution of alum rendered basic by addition of an alkali, continues to grow, preserving at the same time its cubical form; and if its faces be cut so as to make it an octahedron before placing it in such a solution, the cubical shape is in a short time restored. The opposite phenomenon is observed when an octahedral crystal, cut into a cube, is immersed in a solution of ordinary alum.

When the cubical crystals of Roman alum are dissolved in water, at a temperature ranging between 96° and 104° Fahr. (35°·5 to 40° C.), and the solution set aside to crystallize by spontaneous evaporation, the alum is again produced in cubes. At a higher temperature the solution becomes turbid from the separation of basic alum, and the clear liquor deposits alum in octahedral crystals.

The solubility of potassium alum is, according to POGGIALE, as follows:—

Alum melts at 197·6 Fahr. (92° C.).

		Fahr			
100 parts	of water at	32°,	dissolve	3·29	parts of alum.
"	"	50	"	0·52	"
"	"	86	"	22·00	"
"	"	140	"	31·00	"
"	"	158	"	90·00	"
"	"	112	"	357·00	"

At a white heat all the sulphuric acid and the water of crystallization pass off as sulphurous acid, oxygen, and steam, leaving a mixture of alumina and potassium sulphate. At a still higher temperature aluminate of potassium is obtained. Potassium alum, on being heated to redness with sugar out of contact with air, is converted into alumina, carbon, and potassium sulphide, in a fine state of division, and constitutes the pyrophorus of HOMBERG, which ignites spontaneously on exposure to air.

Alum, when heated with alkaline chlorides, liberates hydrochloric acid; again, if a concentrated solution of this salt is boiled with sodium or potassium chlorides, the same acid is given off, and a sparingly soluble basic alum precipitated. RICHTER states that a solution containing alum, chloride of sodium, and nitrate of soda, is a solvent for gold.

When alum is dissolved in twenty parts of water, and ammonia dropped slowly into the solution till the liquid is nearly saturated, a bulky white precipitate appears, which, when thoroughly washed with distilled water, is pure alumina. If dried and weighed, it will be found to be 10·82 per cent. of the weight of the alum taken. If this precipitate while moist be dissolved in dilute sulphuric acid, it constitutes, when as neutral as possible, the normal aluminium sulphate of alumina ($Al_2(SO_4)_3$), which requires only 2 parts of cold water for solution. If this solution be now decomposed by pouring into it liquid ammonia, there appears an insoluble white powder, the tribasic sulphate or basic alum. It contains three times as much earth as the neutral sulphate. However, by adding a strong solution of potassium sulphate to that of the neutral sulphate, a white powder will fall, which is true alum. When recently-precipitated alumina is boiled in a solution of alum, a portion of the earth enters into combination with the salt, constituting the insoluble compound which falls as a white amorphous powder: the same combination occurs if a boiling solution of alum be decomposed by potassium hydrate.

Ammonium alum, ammonio-aluminic sulphate ($Al(NH_4)SO_4.12H_2O$) behaves in all respects as does potassium alum, except that it is more soluble in water, 100 parts dissolving 5·22 parts of ammonium alum at 32° Fahr. (0° C.), and 421·9 parts at 212° Fahr. (100° C.). When heated to redness it loses both its sulphuric acid and ammonia, and becomes alumina. Addition of ammonia to a boiling solution of ammonium alum gives rise to a basic alum as in the case of potassium alum.

Argento-aluminic sulphate, silver alum, has been prepared by CHURCH by heating silver and aluminium sulphates, together with water in an oil bath. This salt crystallizes in octahedra, and is decomposed by water. Its formula is $AlAg(SO_4)_2.12H_2O$.

Sodio-aluminic sulphate, $AlNa(SO_4)_2 12H_2O$, sodium alum, crystallizes in large octahedras, but with great difficulty. But for this cause it would be largely used on account of its great solubility. It is much more soluble than potassium alum; 10 parts of water dissolve 11 parts of this alum at ordinary temperature. In all other characteristics it resembles potassium alum.

The relative solubilities of the three common alums at 60° Fahr. (15°·5 C.) in 100 parts of water are—ammonium alum, 9·37; potassium alum, 14·79; sodium alum, 327·6.

SULPHATE OF ALUMINA. — *Aluminium sulphate*, $Al_2(SO_4)_3 + 18H_2O$.—This substance has of late years been very largely manufactured, and sold under the name of concentrated alum. It occurs in commerce as four-sided plates about an inch in thickness, which are white and partially translucent, dissolve perfectly in water, exhibit no trace of crystallization, and possess the peculiar taste of alum in a more marked degree than even alum itself. The commercial salt is entirely free from iron, and can be cut with a knife. It is very soluble in water, and has an acid reaction. Heated to redness, it is decomposed, leaving pure alumina. The process for its preparation consists in treating clay, cryolite, bauxite, &c., with sulphuric acid in the manner before described; and differs only in adding no potassium sulphate to the solution of aluminium sulphate, and in precipitating any iron that may be present by adding potassium ferrocyanide, evaporating to the consistence at which the mass will solidify when cold, and then pouring it into the "melting vessel." MOHR found this substance, which is improperly called alum, to be perfectly free from iron, and to contain—

Alumina,........................	13·91	per. cent.
Sulphuric acid,....................	36·24	"
Water,..........................	49·60	"
Potassium sulphate,............ ..	1·50	"

The latter ingredient is due to the small portion of potassa naturally existing in all clays. The preceding composition corresponds exactly with the combinations of

7·1 per. cent. of ordinary potassium alum, and
92·9 " of neutral aluminium sulphate.
The formula of which is $Al_2(SO_4)_3 + 18H_2O$.

WIESMANN'S process for the preparation of pure aluminium sulphate consists in agitating the crude solution of sulphate of alumina in a large wooden vessel with potassium ferrocyanide.

The vessel wherein the mixture is made is similar to A, in Fig. 16. After allowing the bulk of the precipitated prussian blue to subside, the supernatant liquor is run off into the wooden vessels, B B B, where it remains until perfectly clear and free from precipitate, after which the clear liquor is treated as has been already described.

The precipitated prussian blue is repeatedly washed with cold water to separate the whole of the aluminium sulphate, and the solutions are drawn off into some of the vessels, B, for the above object.

The prussian blue is now treated with a lie of caustic soda to separate the iron, and thus the same precipitant serves for a long period with very little loss.

The refuse matter which remains contains some sulphuric acid, and resists the solvent action of water.

Two samples of this residuary matter gave the annexed results:—

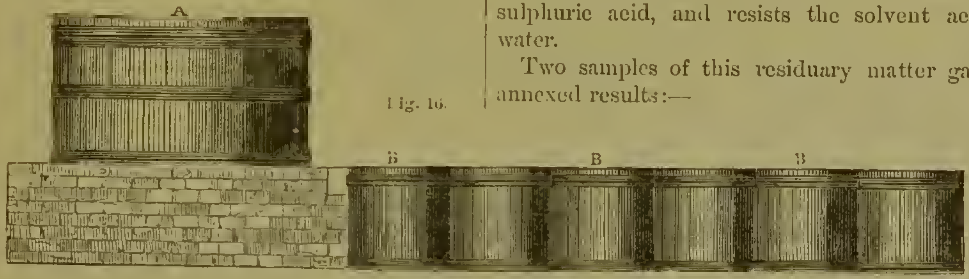


Fig. 16.

Silica,..................................	41·85 ..	39·05
Alumina, with traces of sesquioxide of iron,	18·40 ..	25·55
Sulphuric acid,........................	4·45 ..	8·47
Sulphate of lime,......................	4·01 ..	traces
Water,.................................	29·53 ..	27·50
Loss,..................................	1·76 ..	—
	100·00	100·57

The composition of aluminium sulphate, as met with in commerce, varies very greatly. In four samples analyzed by VARRENTRAPP, he found:—

		Theoretic amount of Sulphuric Acid.
	1.	
Alumina,..................	15·3	
Sulphuric acid,.............	38·0...........	35·8
	2.	
Alumina,..................	12·5	
Sulphuric acid,.............	30·6...........	29·2
	3.	
Alumina,..................	15·1	
Sulphuric acid,.............	38·0...........	43·3
	4.	
Alumina,........	13·0	
Sulphuric acid,.............	34·0...........	30·5

And also that the quantity of water varies even in the same cake.

FLECK found similar differences. Below are given his results for three samples:—

	I.	II.	III.
Aluminium sulphate,...........	47·35	50·80	51·63
Sodium sulphate,..............	4·35	1·24	0·77
Free sulphuric acid,...........	0·73	0·27	—
Water,......................	47·37	47·47	46·94
	99·80	99·78	99·34

In prospect of aluminium sulphate being largely used for the clarification of sewage, Sidney W. Rich devised a process of manufacture by which he states that this salt may be profitably manufactured on the large scale to sell at 30s. per ton. He makes use of the aluminous shale of Guisborough; after burning, and whilst still hot, he places it in a brick tower 18 feet high. By withdrawing the burnt material from the bottom, and continually renewing the heap of burning shale at the top of the tower, a continuous passing of the material down the tower is effected. Whilst this is in progress sulphurous acid gas is introduced at the bottom of the tower from a pyrites burner, mixed with air and steam. Owing to the high temperature, and to the joint presence of the several reagents, the whole of the sulphurous acid gas is converted into sulphuric acid, and in consequence masses of the burnt shale come down, in large part converted into soluble aluminium sulphate. The method of lixiviating employed involves the treatment of the hot crude sulphate with water in tubs which run on an inclined tram, and work on the principle of the lixiviating vats for alkali. The crude sulphate being lixiviated while it is at a high temperature, the liquor becomes boiling hot, and is drawn off in a highly concentrated state; so that very little expense is incurred for fuel and evaporating pans.

Pure aluminium sulphate crystallizes, although with difficulty, in thin pearly flakes, and takes up at the same time 18 molecules of water. It is soluble in half its weight of cold water, but is scarcely at all soluble in alcohol. When heated it parts with its water of crystallization, and leaves a white porous mass, which is anhydrous aluminium sulphate ($Al_2(SO_4)_3$). This modification dissolves very slowly in water.

USES.—Alum is largely employed in the arts, chiefly in consequence of the affinity which alumina has for colouring and other vegetable matters; for gelatine, and a number of like bodies. The fibre of cotton, wool, &c., has such an affinity for alumina or a basic salt of aluminium, that when immersed in a solution of alum a basic salt is formed, which adheres to it so firmly that it cannot be removed by washing.

This basing with aluminium salts enables the cloth to unite with larger quantities of the colouring matter, and retain it with greater persistency. Some precautions are to be observed in its use in the hands of the cloth printer, which will not here be entered into, as they form a part of the subject to be treated of under CALICO-PRINTING.

Alum is used in preparing white leather, technically "tawing;" to clarify water, by the combinations which it forms with the foreign matters present, and also in the preparation of bookbinder's paste. A large quantity of alum is taken medicinally, as a topical and internal remedy in various diseases. Alum mixed with gypsum (calcium sulphate) forms the chief ingredient with which the outer chambers of MILNER'S fire-resisting safes are lined, so as to protect the interior from being injured. This it does from two causes: firstly, the large quantity of water it contains, which helps to moisten the inner chamber when the box is heated, and so prevents the contents from being consumed; and secondly, from the non-conducting power of the mixture after the water is expelled. The great success which has attended the application is the best testimony to its efficiency.

ALUMINA.—*Aluminium Oxide. Alumine*, French; *aluminum oxyd, alaunerde, thonerde*, German. Formula, Al_2O_3.—This body is assumed to be a sesquioxide, from its being isomorphous with ferric oxide (Fe_2O_3).

The utility of alum in calico-printing rests solely upon its being a soluble modification of this earth. Alumina is extensively used, united with silica, in the manufacture of all kinds of earthen and porcelain ware; in the manufacture of crucibles, mortars, and cements. It is particularly employed, almost pure, for the manufacture of *glass pots*, the refractory power of the earth being so great as to permit the fusion of the *frit*, itself remaining unaltered either by the intense heat or by the molten substances.

Alumina occurs in the earth's crust, but rarely in a pure state. When so found it constitutes the mineral corundum, varieties of which, distinguished by their colour, are the sapphire, the ruby, oriental topaz, amethyst, &c.

Alumina constitutes a large proportion of all the slaty and shaly rocks. It is the main ingredient also of pipe-clay and argillaceous soils, which increase in tenacity in proportion to the quantity of alumina they contain.

Though it exists so largely in the soil, it contributes but little in a direct manner to the nourishment of plants, as the ash they leave contains, in general, a very small quantity of alumina.

Emery, much used for polishing and grinding, is an opaque variety of corundum. The Bessemer wheel for grinding steel without loss of temper is composed of emery bound together by RANSOME'S silicate of lime. It thus forms a cutting stone capable of grinding chisels from old files, at a cost of less than 5 per cent. of the stone in comparison with the metal.

Preparation.—Alumina may be precipitated by an alkali from any of its solutions as a bulky, white, amorphous powder, which, when collected, well washed, and dried, is pure. It is a white, tasteless, earthy substance, adheres to the tongue, has a specific gravity of 2·00, and is insoluble in water, but dissolves easily in caustic potassa and soda, and in most acids, at least when newly thrown down. When heated to redness, however, it becomes hard and dense, as in burned clay and firebricks, gives out sparks when struck with steel, and can hardly be scratched with a file. It is then almost insoluble, even in the strongest acids. ROSE states that the specific gravity of alumina increases after long ignition. After exposure for some time in a porcelain furnace alumina has the spec. grav. 3·999, which is very nearly that of native corundum.

Pure alumina is totally infusible at all moderate temperatures; but exposed to that of the oxyhydrogen blowpipe it fuses into transparent globules, which can be made to resemble the ruby by addition of a small quantity of potassium chromate. EBELMEN succeeded in making minute artificial rubies by heating together in a porcelain furnace a mixture of 1 part of alumina with 3 to 4 parts of borax. The alumina dissolves in the borax, and as the latter is evaporated by the intense heat, crystallizes out as corundum. The addition of potassium chromate, as in the previous case, gives to these crystals the colour of the ruby.

Crystallized alumina is insoluble in all acids, but can be rendered soluble by fusion with potassium or sodium hydrates.

Alumina forms three compounds with water:—

Monohydrate,...... $AlHO_2$ or $Al_2O_3 + H_2O$.
Dihydrate,........ $Al_2H_4O_5$ or $Al_2O_3 + 2H_2O$.
Trihydrate,........ $Al_2H_3O_3$ or $Al_2O_3 + 3H_2O$.

The monohydrate is found as the mineral Diaspore. When heated to 360° C. it loses the whole of its water.

The dihydrate was discovered by Walter Crum. It is produced by exposing aluminium diacetate to the temperature of boiling water for several days in a close vessel. The acetic acid appears to be liberated, for on subsequently boiling the liquid in an open vessel, nearly the whole of the acid is given off, leaving the alumina still in solution. This aluminous solution is, however, incapable of acting as a mordant, since it forms with dyewoods transparent lakes, in place of the dense opaque lakes which are desired. On evaporating the solution to dryness the dihydrate is precipitated. Dihydrate of alumina is soluble in acetic acid, but insoluble in sulphuric, hydrochloric, and nitric acids. It is converted into the trihydrate by boiling with a caustic alkali.

Aluminium trihydrate is the gelatinous precipitate produced on treating aluminium salts with alkaline carbonates, ammonia, or ammonium sulphide.

Aluminium trihydrate has a strong affinity for organic matters, and when digested with solutions of vegetable or animal colouring matters, forms with the colours insoluble compounds, termed "lakes."

Trihydrate of aluminium is found native as Gibbsite—a translucent mineral with a fibrous structure, soluble in acids.

ALUMINIUM.—*Alumium*, German; *Aluminium*, French. Symbol, Al. Atomic weight, 27·4.—After having succeeded in isolating the metals of the alkaline earths, Davy attempted, but without success, to separate aluminium from its oxide, alumina. In 1826 Oerstedt formed aluminium chloride by passing chlorine over a mixture of alumina and charcoal, heated to redness in a porcelain tube, and essayed, but in vain, to decompose this salt by potassium and sodium. In 1828 Wöhler repeated Oerstedt's experiment, and by adopting proper precautions to exclude atmospheric air, succeeded in obtaining aluminium, in the form of a grey powder, from its chloride, by igniting the latter with potassium. In 1815 Wöhler prepared the metal in much larger quantity, and obtained it as tin-white globules.

H. St. Clair Deville, in 1854, succeeded in manufacturing sodium on so large a scale, and at such a price, as to render its use available as a reducing agent. By skilful arrangement of apparatus he turned his cheap sodium to account in the preparation of aluminium. He carried out Wöhler's process on a manufacturing scale thus:—

A large iron or copper tube was placed in an unlit combustion furnace constructed for the purpose. Oerstedt's aluminium chloride was then introduced at one extremity of the tube, and at the same extremity a current of dry hydrogen gas was made to enter the tube, and was sustained until the operation was finished. The chloride was now gently warmed by pieces of hot charcoal, in order to drive off any hydrochloric acid which it might contain, and porcelain boats, filled with small lumps of sodium, were inserted in the opposite extremity of the tube. The heat was then rapidly raised until it reached a dull red heat, at which temperature the vapour of the sodium decomposes that of the aluminium chloride. Intense ignition usually attends this reaction. On withdrawing tho porcelain boats the aluminium is found adhering in buttons to the earthenware, mixed with sodium chloride and undecomposed aluminium chloride. The boat was now transferred, with its contents, to a porcelain tube, through which hydrogen gas was passed. At a red heat, the double chloride distilled into a receiving vessel attached to the tube for the purpose; the buttons of aluminium were collected, washed with water, and subsequently fused together under a flux consisting of the double chloride.

Another method of obtaining aluminium was also adopted by H. St. Clair Deville with great success. It was founded on a process originally devised by Bunsen in 1854. By fusing aluminium chloride with an equal equivalent of common salt, he obtained a double chloride ($Al_2Cl_6,2NaCl$), which fused at 360° Fahr. (200° C.). Acting on this body by electrolysis Bunsen isolated aluminium. Deville took—

400 parts of the double chloride of aluminium and sodium.
200 parts of fluor spar or cryolite.
200 parts of sodium chloride.

After carefully drying and mixing, these are laid in alternate layers, with 80 parts of sodium (cut into small pieces), in a crucible lined with alumina, a layer of sodium being put in first. When the crucible is filled a little powdered salt is to be sprinkled on its contents, and the crucible, fitted with a lid, is introduced into a furnace until the reaction, whose occurrence and continuance is indicated by a peculiar and characteristic sound, shall have terminated. The contents of the crucible, having been stirred with a porcelain rod while in their liquefied state (this part of the operation is essential), are poured out on a surface of baked clay or any other suitable material, the flux, &c., on one side, and the metal on the other. The cryolite here chiefly plays the part of a flux; but in 1855 Dr. Percy obtained aluminium directly from this mineral.

When the process is conducted on a large scale, the mixture is heated on the floor of a reverberatory furnace. The proportions used being, sodio-aluminic chloride, 10 parts; cryolite, 5 parts; and sodium, 2 parts. The chloride and cryolite are powdered and mixed with small lumps of sodium, the whole being then thrown into the already heated furnace, and the doors and dampers closely shut to exclude air. The action speedily takes place, and becomes so intense that tho walls and hearth of the furnace are raised to bright redness, and the mixture itself becomes almost entirely liquid. When the operation is complete, the furnace is tapped at the back, the slag flows out first, and at the last the aluminium. The first portions of slag consist almost entirely of aluminium fluoride (Al_2F_6), and contains minute globules of aluminium, to extract which it is pulverized and passed through a sieve. The fluoride may be used to prepare aluminium.

Cryolite, *i.e.*, aluminium and sodium fluoride ($Al_2F_6.6NaF$), occurs in great abundance at Evijtok in Greenland. It is now very largely used for the preparation of aluminium. The mineral is powdered, mixed with half its weight of common salt, and either placed in alternate layers with 2 parts of sodium in a crucible, or roasted with the same quantity of sodium in a furnace.

The chief advantage in using cryolite is, that the costly and troublesome process of preparing the double chloride of aluminium and sodium is thus obviated. The metal is, however, less pure. Many patents have been taken out for improved processes, but they all apply to mechanical contrivances for economizing the sodium.

GERHARD proposed to decompose aluminium fluoride or cryolite by a current of hydrogen at a red heat. The aluminium fluoride, or cryolite, was placed in shallow earthenware dishes, alongside of which were like dishes containing iron filings, in an oven which had previously been heated to redness. A current of hydrogen was then admitted and the heat increased. Hydrofluoric acid gas was given off and absorbed by the iron filings. This process was, however, ultimately abandoned by its inventor, who reverted to the use of sodium.

No processes of aluminium smelting, except those of reduction by sodium, have been thoroughly successful. The progress of the manufacture of aluminium therefore depends on still more economical methods for producing the alkaline metal being invented. Mr. J. LOTHIAN BELL, of Newcastle-on-Tyne, manufactured aluminium on the large scale for several years, but has of late relinquished the undertaking on account of the limited market for the metal.

PROPERTIES.—The physical properties of aluminium are very characteristic. Its specific gravity is 2·56; after hammering, 2·57. This low specific gravity, being nearly that of the flux employed in fusing it, materially enhances the difficulty of its production. Aluminium is next highest in density to the metals of the alkaline earths. It is malleable, ductile, and sonorous. A bar of aluminium, when struck, emits a clear musical ring; when cast into the shape of a bell and struck the sound is, however, anything but musical; it more resembles that of a cracked pot. The fusing point of aluminium is intermediate between silver and zinc, but nearer to the latter. It resembles silver in its excellence as a conductor for electricity. Its capacity for heat is very great, about six times that of silver. Its chemical characteristics are such as could not have been predicted. Instead of reassuming oxygen, like the metals of the alkalies and alkaline earths, with an energy proportioned to the extreme tenacity of that element when in the state of oxide, aluminium is almost as indifferent to oxygen as are gold and platinum. It is not affected by sulphur as is silver and lead, nor is it acted on, except very slowly, by nitric and sulphuric acids in the cold. Its only solvents are hydrochloric acid and aqueous solutions of potassium and sodium hydrates. The strong affinity between this metal and oxygen before its separation, contrasted with its apparent total indifference afterwards, suggests the possibility that, at the instant of its coming in contact with air, aluminium may receive a fine coating of oxide, a film of transparent sapphire, from the atmosphere, which protects it from all further action. This conjecture is rendered probable by the result obtained on exposing a leaf of aluminium to the oxidizing flame of the blowpipe. The result of the combustion, though apparently a mass of alumina, shows by its metallic lustre, when rubbed in an agate mortar, that the oxygen has not penetrated below the surface.

ALLOYS.—Aluminium forms alloys with nearly all metals. With zinc and iron it forms brittle compounds. With silver it also forms a brittle alloy, unless the proportion of silver be very small—3 to 5 per cent. Silver alloys are used for making many ornamental articles, and are said not to be tarnished by sulphuretted hydrogen.

Aluminium Bronze.—This is the most important of the aluminium alloys. It is a definite compound of copper and aluminium, which has the formula Cu_9Al. This substance is somewhat largely used as a substitute for gold, which it closely resembles in colour. It takes a high polish, is very malleable though hard, and possesses a tenacity equal to that of steel.

AMMONIA. — *Ammoniacal Gas.* — *Ammoniaque*, French; *Ammoniak*, German. Ammonia is synonymous with the *alkaline air* of PRIESTLEY, *volatile alkali*, &c. Formula, NH_3.

Gaseous ammonia was probably known to the ancients, though no record of the fact remains to prove it; still, as the manufacture of ammoniacal salts was a source of trade among the Egyptians, it is very probable the pungency of the gas was familiar to them. Ammonia was noticed by RAYMOND LULLY in the thirteenth century, who prepared it from urine; the name he gave to it was *mercurius vel spiritus animalis.* BASIL VALENTINE, in the fifteenth century, separated ammonia from sal-ammoniac; he termed it *spiritus urinæ.*

The scientific development of this compound remained for Dr. BLACK, who in 1756 first isolated the gas, and proved its distinction from the sesquicarbonate, $(NH_4)_4H_2(CO_3)_3$; PRIESTLEY afterwards, in 1774, with greater accuracy, in a more lengthened investigation, prepared it in a pure state, and discovered more of its properties, and noticed its decomposition by the electric spark. SCHEELE, in 1777, discovered that ammonia contained nitrogen. BERGMANN gave it its present name, ammonia, in 1782; and its true chemical composition was worked out by BERTHOLLET in 1785, and corroborated by his son in 1808.

Ammonia, though not very extensively disseminated in nature, is to be met with in a great many vegetable products; the decomposition of animal matter, either by putrefactive fermentation or destructive distillation, yields it in moderate abundance; it is also found united with acids in many strata of the mineral kingdom. In the atmosphere, very minute portions of ammonia are always present, which exert a most beneficial influence upon vegetation. Volcanic districts are highly productive of ammonia, standing second in this respect only to the guano islands of South Africa. Sea water contains a low decimal percentage of ammoniacal salts; fresh water also holds it in combination, but in an infinitesimal proportion. A perceptible quantity of this alkali, generally combined with acids, is found in mineral springs; soils almost universally contain it—more especially those of the ferruginous and argillaceous class. In many substances where ammonia is apparently absent, a careful and patient investigation may prove its presence. Thus, it occurs in the atmosphere; apparently no ammonia exists in it when performing an analysis, but on examining rain water recently collected in town or country, an expert chemist will always detect it in minute quantity.

Animal excrements, especially those of reptiles, are rich in ammoniacal compounds.

Preparation.—It may be prepared in the laboratory by a very simple process, namely, by heating an ammoniacal salt with quicklime in a retort, and collecting the eliminated alkaline vapour over mercury, or by displacement of air. This operation, though simple, requires—like all those conducted on the small scale—care and skill to obtain a pure product. The decomposition which takes place in the preceding instance, supposing that ammonium chloride had been employed, is the following:—

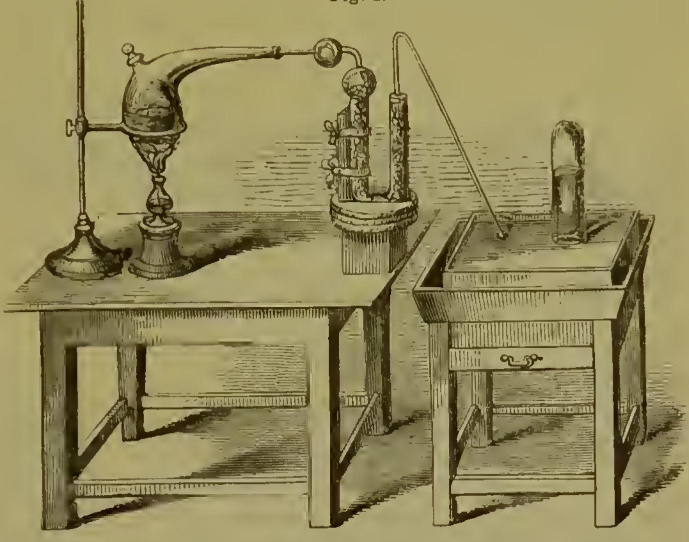
$$\overset{\text{Ammonium Chloride.}}{2NH_4Cl} + \overset{\text{Lime.}}{CaO} = \overset{\text{Ammonia.}}{2NH_3} + \overset{\text{Calcium Chloride.}}{CaCl_2} + \overset{\text{Water.}}{H_2O}.$$

The arrangement in Fig. 1 is well adapted for the preparation of ammoniacal gas: equal weights of the ammonium chloride and quicklime which has been just slaked are intimately mixed, and introduced into the retort, heat is applied, gently at first, and then gradually increased till the gas ceases to be evolved—calcium chloride remains.

One volume of nitrogen and three volumes of hydrogen cannot be made to unite directly, even when passed over red-hot spongy platinum. But on igniting a mixture of hydrogen (in excess), nitrogen, and oxygen, ammonium nitrate is formed.

In a chemical point of view, ammonia is generated in various and curious ways; as, when an excess of hydrogen is burned in the atmosphere, nitrate of ammonia is formed; when iron oxidizes, from the decomposition of water, sesquioxide of iron and ammonia result. A similar combination is formed when hydrates of potassium, sodium, barium, or calcium, are heated in the air, or in hydrogen, with iron, zinc, lead, tin, or arsenic. According to Faraday, this combination takes place even in an atmosphere of hydrogen. Reiset explains its formation in hydrogen as arising from nitric oxide derived from the sulphuric acid used to prepare the hydrogen, and adduces as a corroboration the fact that when iron filings are heated in a strong solution of potassium hydrate, they evolve hydrogen and ammonia, both when heated in air and in hydrogen containing nitric acid, but not in pure hydrogen. When nitrous oxide and an excess of hydrogen are transmitted over platinum black—spongy platinum—in the cold, no ammonia is formed; but if heat be applied it is produced in considerable quantity. A dilute solution of nitro-sulphuric acid—composed of 100 volumes of water, 4½ of sulphuric acid, and 4 of nitric acid—dissolves iron, zinc, or tin, without giving off nitric oxide or hydrogen, although these bodies always result from the decomposition of nitric acid and the solution of the forementioned metals; but in this instance the nascent hydrogen combines with the nitrogen of the nitric oxide, and gives rise to ammonia.

Fig. 1.



Ammonia is a volatile, irrespirable gas, though when diluted with air it may be inhaled. Its odour is extremely pungent; it possesses a strong alkaline taste; turns reddened litmus paper blue, and turmeric paper brown; is very slightly inflammable, and extinguishes those bodies which are in a state of combustion. It is composed of two volumes of nitrogen and six of hydrogen, condensed into four volumes of the gas. The specific gravity of the gas is ·5893, air being taken as unity.

By a pressure of 6½ atmospheres, at the ordinary temperature, it is condensed into a transparent colourless liquid, of 0·731 specific gravity at 60° Fahr. (15°·5 C.); a like result is obtained at the ordinary pressure of the atmosphere, by reducing the temperature 40° Fahr. below zero (—39°·9 C.). It is a very subtle, colourless liquid, which, according to Faraday, freezes into a white translucent crystalline substance at —108° Fahr. (—57°·1 C.). On heating one volume of the liquid to 60° Fahr.

(15°·5 C.), at a barometric pressure of 30·2 inches, it formed 1009·8 volumes of the vapour.

FARADAY succeeded in obtaining ammonia in the solid state by exposing the gas to the pressure of 20 atmospheres, whilst reduced in temperature by surrounding the vessel with a mixture of solid carbonic acid and ether.

Ammonia is decomposed with facility by heat in the presence of any substance which contains oxygen easily transferable; hence, if the higher oxidized compounds of nitrogen be brought into contact with ammonia, and both compounds are mutually decomposed, water and a lower oxide, with the occasional elimination of nitrogen, are the result. When transmitted over many metallic oxides at a dull red heat, it is decomposed into nitrogen and hydrogen; the latter abstracting oxygen from the oxide and forming water. It is also split up into its constituent gases by a succession of electric sparks.

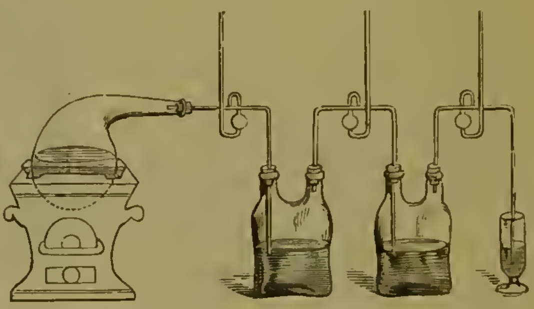
LIQUID AMMONIA.—*Aqueous Ammonia. Salmiakgeist*, German; *esprit de sal ammoniac*, French; synonymons with *caustic ammonia* and *spirit of hartshorn*. Formula, NH_4O.—The preparation of liquid ammonia is one among the many manufactures which have sprung up in consequence of the rapid progress of science and art during the last half century; but it seems to have risen to its present state simultaneously with that of gas-lighting. Considerable quantities of crude liquor ammonia are produced as one of the by-products of the distillation of coal; hence the establishment of factories for the preparation of ammonia and its salts has gradually taken place, from the metropolis to almost every provincial village where gas is consumed. The demand for animal charcoal opened another extensive supply of ammonia, for it is evolved during the destructive distillation of the bones from which the animal charcoal is prepared for the sugar refiner and others; and as soon as the spirit of enterprise and emolument induced the capitalist to turn his attention to the manufacture, various other sources were discovered which yielded this alkali. The principle on which ammonia is generated on the large scale differs in nothing from the processes already stated; and the formation of the solution of the alkali rests upon the property which water possesses of absorbing it. Water, at a temperature of 50° Fahr. (10° C.), condenses about 670 times its bulk of ammoniacal gas, DAVY (*i.e.*, nearly half its weight), 780 according to THOMSON; by this retention the bulk of the solution is increased considerably, so much so, that 6 volumes of water, on becoming saturated with the gas, increase to 10 volumes, and the specific gravity of the liquid is reduced from 1·00 to 0·875.

Liquor ammonia is prepared in the laboratory by taking a mixture of 1 part of sal ammoniac with 1¼ part of slaked lime, and 1½ part of water, and introducing it into a glass or earthenware retort, the neck of which is connected with a series of Woulfe's bottles, described at page 78—one of which contains a little water to arrest impurities; the second, a weight of water equal to that of the ammonium chloride used, space being allowed for expansion of the liquid; a third is useful to stop the uncondensed ammonia which would otherwise escape into the air. The ammoniacal solution in the last two bottles is pure. It is necessary that the lime used should be slaked in about 4 parts of water, previous to its being mixed with the powdered ammoniacal salt, as by that means the ammonia will be more freely disengaged, and with less heat than when only a small quantity of moisture is present.

Fig. 2 is a representation of an apparatus which answers the purpose remarkably well.

During the decomposition the heat should not be too briskly applied, and in no case should the bottom of the retort be raised to redness, as portions of the salt would be sublimed without being decomposed; and by passing over to the first bottle, would render its contents unfit for use. To guard as much as possible against the sublimation of the salt, the tube connecting the retort and first bottle should have a considerable calibre; and it is often advantageous to append a large bulb or balloon between the mouth of the retort and first condenser, in order that any portion of the sublimed salt which might be driven off towards the end of the operation, may be arrested before it is carried into the ammoniacal liquor. The greatest precaution is to be taken in having all the connections air-tight, to prevent the escape of vapour. When the whole of the ammonia is driven off, it is necessary to raise the temperature a little higher than usual to fuse the chloride of calcium; and as soon as this takes place, the retort should be disjoined from the condensing bottles, and the fused salt poured out as speedily as possible. Very often the retort breaks at this part of the operation, in consequence of a layer of the salt solidifying upon its neck, which on being exposed to the air readily absorbs moisture, and partly dissolves; the solution thus formed cools considerably, and on penetrating through the undissolved portion of chloride of calcium to the retort, which still retains a high degree of heat, the particles with which it comes in contact are suddenly contracted, and the retort cracks. Should the retort escape fracture at this part of the process, it generally happens that it is broken when the heat is applied for a second operation; hence it is rare to find a retort, even when carefully handled, that will stand two operations.

Fig. 2.



Uses of Ammonia.—Ammonia is in daily requisition as a reagent for the analytical chemist. It offers peculiar facilities in the preparation of many compounds on account of its great volatility, as also that of its salts, and as being an almost universal precipitant of the oxides of the heavy metals. In medicine, ammonia is used to a moderate extent to alleviate spasms, for rousing the vascular and respiratory system, as an antacid, and in various other cases. It is extensively employed in bleaching and calico-printing, in colour manufactories, for making artificial manures, and other important trades.

When ammonia is manufactured on the large scale from ammoniacal salts, the sulphate of the alkali is for the most part preferred; but there is no material deviation from the directions already laid down, except that another kind of apparatus is used, to avoid the loss of retorts which is incurred when preparing it in the laboratory. It is particularly necessary, when working upon the large scale with condensers in the form of Woulfe's bottles, to have the joints well luted; this is effected by covering each connection with a paste made of white of egg and chalk intimately ground. A better composition is produced by blending wax, resin, and turpentine together; this should be applied upon the connection of the tubes and tubulures of the condensers in moderate thickness. If this point is not attended to with care, the pressure upon the gas forces it through into the atmosphere, causing the workmen considerable annoyance till arrested.

Fig. 3.

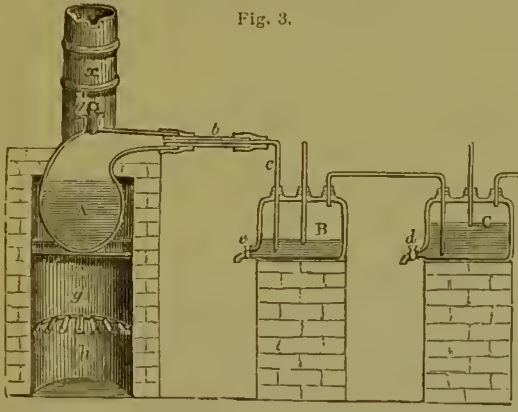


Fig. 3 is a vertical section of an apparatus which answers well for the preparation of ammonia on a large scale.

A is an iron retort, placed in sand, over the fire *g*, of which *h* is the ash-pit, and *x* the chimney; *a* is the stopper, and *b* an iron pipe connected to the neck of the retort, and reaching some distance from the furnace to the end of the tube, *c*, which may be either of glass or lead. B is the first condensing vessel, and is supplied with three tubulures, through the middle one of which a safety tube is inserted. *e* is a stopcock and pipe, by which the contents of B are drawn off when requisite. This vessel is left nearly empty, for the purpose of purifying the gas of any traces of ammoniacal salt which may be carried over, and also for retaining traces of oily matters which are invariably present, either from the impurity of the salt employed, or from the grease with which the stopper is smeared to prevent it from adhering too tightly in its place. B is connected with other condensers, of which only one, C, is seen in the figure; these vessels are filled to about three-fourths of their capacity with water, and so rapid is the absorption of the gas, that scarcely a trace of ammonia escapes from C till the solution contained in it is completely saturated.

When this happens, the solution of ammonia is drawn off by the stopcock, *d*, and the very weak liquor in the third condenser supplied instead, or it is recharged with a fresh quantity of water.

In this process it is very easy to produce, at each operation, an ammoniacal liquor of any standard strength, by furnishing each condenser with a glass gauge pipe, graduated into equal parts, showing the bulk of liquid in the interior. Whatever be the quantity of water introduced, it expands in the ratio of six to ten, as before stated, on being completely saturated with the alkaline vapour, or in less proportion according as its gravity is reduced. At the first working, the measure of water employed is noted, and also the proportion of expansion, till it approaches that point which, by calculation, is found to correspond with the strength of the liquid required; this may be corroborated by drawing off portions repeatedly, and taking their density, and from this number the quantity of ammonia is ascertained by the aid of tables, which will be found further on. As soon as the exact point is gained, the gauge is scratched at the level of the liquid, and this one test will serve for all future operations where the ammonia is to be made of the same strength; but it is necessary that the bulk of liquid should be always equal to that used in the first experiment.

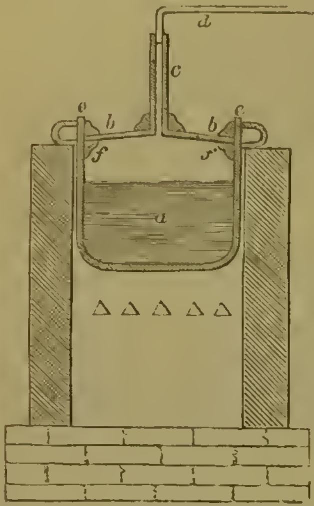
When gas ceases to be evolved from the mixture in the retort, the fire is partly urged, and the stopper removed by means of a lever. If the stopper be so firmly fixed that it cannot be readily displaced, a cloth moistened with cold water should be carefully wrapped round it, without touching the neck of the retort; this refrigeration causes a contraction in its particles, and will enable the operator to remove it with facility. The residue—which is fused chloride of calcium, in the event of the chloride of ammonium being used—is then ladled out. Should sulphate of ammonia be operated upon considerable quantities of water are employed, and the distillation should never be allowed to proceed to dryness, as in such a case the sulphate of lime formed would constitute a concrete on the bottom of the retort, which could not be removed without much labour and loss of time.

All the ammoniacal impurities which have collected in B during the first distillation are drawn off, and introduced into the retort in the second operation, to obtain the ammonia which they hold, and a small quantity of fresh water is substituted, to cover the lower end of the safety tube, as in the preceding operation. This apparatus, when once erected, will last for a long time.

A modification of the foregoing apparatus represented in the annexed cut—Fig. 4—is sometimes employed.

a is an iron kettle, to which a metallic lid, *b b*, is adapted, resting upon blocks or ledges, *f f*, in the interior of the vessel. The lid is rendered air-tight by means of other bars of metal, *e e*, which are soldered fast on the outside over the interior ones. From the middle of the cover an iron pipe, *c*, rises, to the orifice of which another pipe, *d*, bent at right angles, is soldered, for the purpose of conducting the ammoniacal vapour to the condensers, which are similar to those already described. A much larger quantity of water must be used in this apparatus than in the preceding one; and as distillation advances, it is necessary to keep up the supply throughout the process, that the solder may not melt, a circumstance which would happen were this precaution overlooked.

Fig. 4.



As the demand for ammonia and its salts became greater, its manufacture received an additional impetus, and many patents have been granted for its preparation; most of them, however, relate to the production of the salts of ammonia.

Mr. Young, in 1841, took out a patent for preparing ammonia from guano; the method which he recommends is the annexed:—The retorts are filled vertically with two parts by weight of guano, and one part by weight of hydrate of lime, or other caustic alkali, and the whole is intimately mixed by the aid of an agitator placed in the retort. Having well mingled the materials, the retorts are moderately heated, and this heat is gradually increased to redness. The combined action of the heat and alkaline compounds disengages all the ammonia, whether it be in the form of salts or other complex bodies, such as urea or uric acid, and the gas thus liberated is received into a condenser filled with water. Other gases and fluids are evolved together with the ammoniacal vapour, the uncondensable portions of which pass through the condenser unaffected.

An ammoniacal solution is obtained by the destructive distillation of bituminous schist, by Count de Hompesch, who rendered it available for the manufacture of ammoniacal salts.

The method adopted by Watson for manufacturing ammoniacal liquor from gas liquor is as follows:—The gas liquor is run into a capacious retort, and a suitable quantity of slaked lime added—the amount being determined by the quality of the crude liquor; heat is then applied, and ammonia, tolerably pure, distils over, which, on being received in a vessel of cold water, forms an ammoniacal liquor. When considerable quantities of steam are observed to pass over with the vapour of ammonia, the strong alkaline solution already formed is removed; that which is collected afterwards, by continuing the distillation, is weak or impure, and is returned to the boiler, with a second charge of lime and crude liquor, to undergo another distillation. The first portion from the previous operation is introduced into the retort, mixed with a small quantity of lime if necessary, and as soon as the disengaged vapour carries steam with it, the strong liquor in the condenser is to be drawn off as before. The distillation is continued as long as it yields ammonia, and this secondary product is returned to the retort as before. The first portion of the second distillation is a concentrated liquid of sufficient purity for all ordinary purposes of scouring, cleaning, &c., but it may be still further improved by distilling a third time, in the same way as already directed, observing that the portion of solution which is made by the absorption of the ammoniacal gas is preserved, and the residue transferred again to the retort.

A great step in advance in the manufacture of ammonia on the large scale, was made by Newton in 1841; it consisted in the application of Coffey's still, described at page 80, but with a few modifications to adapt it to the distillation of the gas water. By its use the ammonia may be produced of any density up to its most concentrated state, and consequently of corresponding purity. Having made the reader already acquainted with the still in its application to the whisky trade, it will be unnecessary to enter into lengthened details; those specific alterations which are peculiarly required by the operation will, however, be pointed out. The number of diaphragms in the apparatus is increased or diminished according to the strength of the product which is to be produced, and the whole apparatus may be constructed of wood lined with sheet lead, having the diaphragm plates in the interior of the analyzing column formed of perforated sheet-iron. Each of the sheets is supplied with several small valves, so weighted as to open upwards whenever the elastic vapour below the plate exerts more than a certain amount of pressure upon them. The ammoniacal crude liquid, which enters at the top, passes downwards through each diaphragm successively, by means of a pipe rising about 1 inch above the level of the plate, the lower end of which is encased in a cup fixed upon the next diaphragm. The pipe must be sufficiently capacious to carry off all the liquid which enters at the top; and as the solution descends it fills the small cup, and prevents the vapour from ascending at this part of the plate. Steam, as usual, is the medium which effects the elimination of the volatile body. According as the ammonia is to be procured, the supply of liquor and the entrance of steam beneath are regulated. When it is necessary that the ammonia should depart from the top of the rectifying column in the form of gas, the flow of liquid should be in proportion to the ascending steam, in order that the liquor in the top diaphragms may remain at little more than the normal temperature of the atmosphere, but become hotter as it falls towards the under reservoir, where it is retained for some hours at a boiling heat, to expel all the alkali. On the contrary, when it is necessary that the ammoniacal vapour should be in company with much steam, the stream of liquid entering is elevated to nearly its boiling point in the top diaphragms, by means of the larger volume of steam which is admitted from the boiler. Concentrated or dilute solutions of ammonia may in this way be obtained at once from salts, the acids of which are removed in the usual manner by lime or alkali; a product of considerable purity is also derived from the ammoniacal liquids formed by the distillation of bones or other animal matters; but before submitting such liquids to the action of the heat, it is necessary to remove most of the impurities and the combined acid by the action of lime.

It is customary to have the diaphragms bent upwards, alternately, at opposite sides, so as to conduct the liquid from right to left, and conversely, till it reaches the bottom.

Carbonic acid gas, and other volatile compounds which do not impair the use of ammonia in certain applications, may in using this still be given off with the ammonia; but in this case as much steam should be generated as will prevent the absorption of the ammonia gas by the carbonic acid.

LAMING recommends that the solution of ammonium carbonate, as obtained from bones, should be decomposed by means of chloride of calcium, instead of the mineral acids which are usually employed for that purpose; carbonate of lime is formed, and chloride of ammonium remains in solution. After filtering or siphoning this liquid, he boils it for an hour, by which treatment any gaseous impurities are expelled. As soon as the liquid has cooled, it is agitated, first with sufficient hydrated oxide of iron to precipitate any hydrogen sulphide (H_2S) that may still remain, and next with enough lime to saturate the whole of the combined hydrochloric acid; it is then distilled. By this treatment a tolerably pure solution is produced.

It is to be observed, that when ammonia is prepared on the large scale according to the method first described, the dry salt and lime are often mixed in the retort, and as operations are about to commence, a quantity of water is added for the purpose of slaking the lime, and preventing the mass adhering to the bottom of the retort; by this means so much heat is generated that a considerable quantity of the ammonia is driven over to the receivers, and thus fuel is economized. To prevent the residue hardening when the distillation is carried to dryness, it is customary to add a few spadefuls of common salt to the mass.

An ingenious apparatus, which may be taken as typical of that now commonly employed for extraction of ammonia from gas liquor, &c., was recently devised and patented by ELWERT and PACK. The form of apparatus will be readily understood by the following description referring to the accompanying drawings. (Figs. 5, 6, and 7). The same letters refer to the same parts in each figure.

A vessel capable of holding the charge of ammoniacal liquor is so fixed that it may receive the liquors from the tank or reservoir of crude ammoniacal liquor. In this vessel the gas liquor is mixed with the proper quantity of lime, and then run into a boiler.

On the application of the fire to the boiler the ammoniacal vapour as it escapes is led by a pipe back into the first vessel, and allowed to escape into the liquid at its lowest portion; as the gas rises it absorbs fresh portions of ammonia, and thus enriched by the ammoniacal liquor, is led into a condensing chamber, the more condensible portion running back into the first vessel. The more volatile vapour is carried through a refrigerating worm to a condensing tank where the hydrocarbons, some free ammonia, and sal-ammoniac is retained. The purer and uncondensed vapour passing on is conducted into vertical purifiers filled with charcoal, and thence into the stock condensing chamber.

On the dome or upper part of the wrought-iron boiler A is adapted the tube *a*, *a*, which passing upwards for a certain distance is then turned downwards, and enters through a tight joint into the vessel A_1 nearly to the bottom; it is then carried along nearly the whole length of A_1, and is pierced with numerous holes for the escape of the gas passing from the boiler A into this pipe or tube *a*, *a*.

On the dome of the vessel A_1 is adapted the tube *b*, *b*, turned down as a siphon, the other branch going to within 12 inches of the bottom of the vessel B, which is in communication with A_1 by the tube *l*.

On the cover of the vase B is fixed the tube *c*, which leads the aqueous ammoniacal vapours into the coil of the refrigerator C. The tube *c*, when it leaves the refrigerator, is carried to within 8 inches of the bottom of the vessel D, fitted with a safety tube *d*, and a cock *s*, opening a communication by the tube *p* between the vessel D and the boiler A_1.

In the vessel D are deposited the aqueous ammoniacal vapours condensed by the worm, whilst the ammoniacal vapour passes by the tube *e* to traverse four cast-iron tubes, E, E_1, E_2, E_3, communicating by the tubes f, f_1, f_2; these four tubes, open at each end, are fitted with rims so that each end may be closed with a tight fitting cast-iron cover.

From the tube E_3 the ammoniacal vapours are carried by the leaden tube *g* into a lead condensing cistern G, fitted with a funnel-shaped safety tube *h* and a cock for emptying at *t*; the bent tube *i* takes the non-condensed gas in G into the vessel H, which is also fitted with a tap for emptying a funnel-shaped safety tube *j*, and a tube *k* for the last uncondensible portions of the gas; the latter should be led away from the factory.

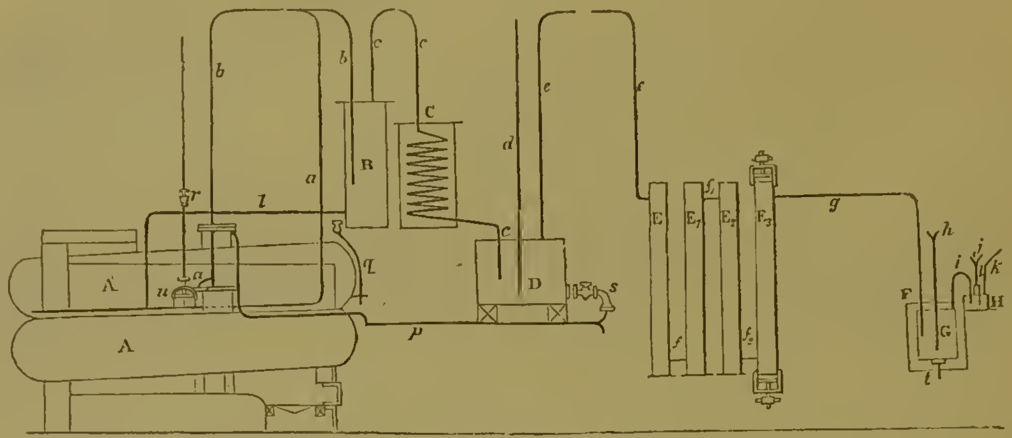
The crude ammoniacal liquors are introduced by the tube and cock *q*, which is in communication with a reservoir above the level of the tank A_1. The capacity of the boiler A_1 is about 300 gallons. The milk of lime is thrown in hot through the hole *r*.

The emptying of the vessel A_1 into the boiler A is effected by the tube and valve *m*, to clear the tube *m* of any matter which might choke it. The rod *o* is fitted in a stuffing box, which allows it to be passed along the pipe without loss of any vapour or liquor. The boiler A is emptied by the tube and valve *n*.

To prevent the formation of vacuum the boiler A is fitted on the tube *a*, *a*, with a small tube in connection with the vessel *u*, which latter carries the safety tube and cock *v*.

The vessel G is placed in a reservoir made of sheet iron, F, containing cold water or a cooling mixture, as the vessel G becomes otherwise too warm by the condensation of the ammoniacal vapour into the water.

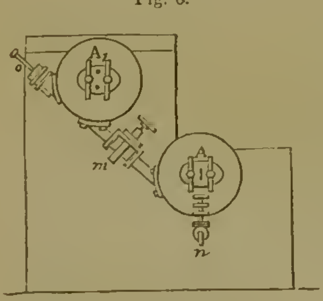
Fig 5.



On the face of the boiler A, about 60 centimètres from the bottom, a small hole is made which is closed with a wooden peg, by means of which it can be ascertained when the vapour of ammonia has been completely driven off.

The mode of working the apparatus is as follows:—The man-holes of the boilers being closed, the valve *n* of the boiler A is closed, the valve *m* is opened, the refrigerators C, F, are filled with cold water, the four tubes E, E$_1$, E$_2$, E$_3$, are packed or filled with freshly burnt charcoal, the covers screwed on and luted with linseed meal. In the vessel G is placed 8 or 10 gallons of pure water, and the vessel H is also filled with water through *j*.

Fig. 6.

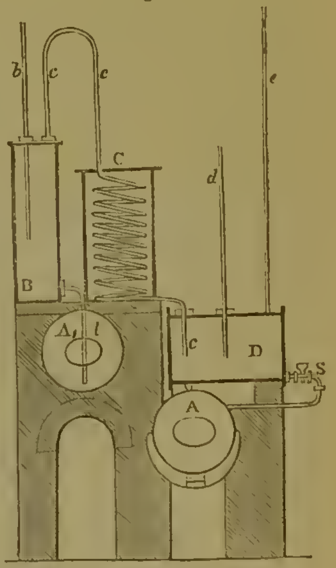


After these preparations the cock *q* being opened, 300 gallons of crude ammoniacal liquor is run into the vessel A$_1$, and at the same time by the hole *r* enough milk of lime to set free all the ammonia in the charge.

When the crude liquor mixed with the milk of lime has run into the boiler A, the cock *v*, the valve *m*, and the hole *r* are closed, and the fire is lighted, the heat from which passing under the first half of the boiler A returns under the other half in its passage to the chimney.

As soon as the contents of the boiler begin to boil the air is expelled, and bubbles through the vessel G and H: the boiler A$_1$ and the tubes *b* and *c* become hot; the air has then been expelled and it is time to charge the boiler A$_1$. The cock *q* is then opened, the vapour is cooled by the liquor running into A$_1$ and produces a vacuum, which draws the surplus liquid in D along the tubes *c* and *p* into the upper vessel A$_1$, at the same time that the air enters by the tube *d*. When A$_1$ is charged the cock *q* is closed, and the cock *v* opened, and through the hole *r* the charge of milk of lime is added, the contents of A$_1$ are well stirred with a rod, the hole *r* closed, and the fire made active.

Fig. 7.



The aqueous ammoniacal vapour of the boiler A, escaping by the small apertures in the horizontal part of the pipe *a*, *a*, agitates the liquor in A$_1$ and rises through the liquid much richer in ammoniacal vapour. This vapour, passing by the tube *b* into the vessel B, partially condenses and loses its aqueous vapour, which with some of the ammoniacal salts deposits in the vessel, from whence it flows back to A$_1$ by the tube *l*; the concentrated vapour passes by the tube *c* into the worm, where the aqueous vapour is entirely condensed, together with a portion of the hydrocarbons, free ammonia, and ammoniacal salts. The liquid accumulating in the vessel D assists towards the end of the operation, when the vapours are more charged with ammoniacal salts, to wash these vapours and retain the salts. The uncondensed vapours pass by the tube *c* to the charcoal purifiers E, E_1, E_2, E_3; the charcoal absorbs all the hydrocarbons, and pure ammoniacal gas escapes from the last purifier by the pipe *g* into the vessel G, where the pure water absorbs the vapour till the liquid has acquired the desired strength. The atmospheric air escapes by the tube *i* into the liquid in the vessel H, which absorbs the last traces of ammoniacal vapour, and passes by the tube *h* outside the factory.

During the operation the refrigerators C and F must be kept constantly cooled.

When the liquor in the boiler A contains no more ammonia it is run off by the valve *n*; and A is again charged from A_1, the cock *v* shut as before, and the crude charge let into A_1 again. The aspiration draws the liquid from the vessel D by the tube *p* and the tube *c*, thus clearing away any deposit in the tubes every time the boiler is charged.

During the time A_1 is being charged the charcoal purifiers are repacked, the liquid ammonia is drawn off from the vessels G and H, and replaced with pure water as before. If the water used in G and H is not distilled water, the ammonia liquor when drawn from G and H must be set aside to deposit the salts of calcium, aluminium, and magnesium; but with pure water the charge is ready for immediate delivery.

The time occupied in the operation is from four to five hours.

Liquid ammonia is of different strengths, according to the more or less complete saturation of water with the gas. As the quality of the solution depends mainly upon the available ammonia it contains, it is the duty of the manufacturer, as well as of the purchaser, to be acquainted with its amount. Specific gravity tables have been constructed by various chemists, for the purpose of ascertaining the value of ammoniacal solutions. In the formation of these tables a great many experiments were performed with liquids of various densities, for the purpose of arriving at their content of ammonia, and the amount corresponding to the intermediate gravities calculated according to the known properties of this alkali.

Of these, the results of DALTON, DAVY, and URE are generally preferred. According to DAVY, the appended table expresses the percentage in every hundred parts of liquid:—

Specific gravity of the liquid.	Percentage amount of ammonia.	Specific gravity of the liquid.	Percentage amount of ammonia.
·8750	32·50	0·9435	14·53
·8875	29·25	·9476	13·46
·9000	26·00	·9513	12·40
·9054	25·37	·9545	11·56
·9166	22·07	·9573	10·85
·9255	19·54	·9597	10·17
·9326	17·52	·9619	9·60
·9385	15·88	·9692	9·50

DALTON attaches the boiling point and the volume of vapour in the mixture:—

Specific gravity.	Grains of ammonia in a hundred of the liquid.	Boiling points.	Volumes of gas in one volume of the solution.
0·850	35·3	26°	494
·860	32·6	38	456
·870	29·9	50	419
·880	27·3	62	382
·890	24·7	74	346
·900	22·2	86	311
·910	19·8	98	277
·920	17·4	110	244
·930	15·1	122	211
·940	12·8	134	180
·950	10·5	146	147
·960	8·3	158	116
·970	6·2	173	87
·980	4·1	187	57
·990	2·0	196	28

The following is the table of the strength of caustic ammonia compiled by URE:—

Specific gravity.	Per cent. of ammonia.	Specific gravity.	Per cent. of ammonia.
0·8914	27·940	0·9363	15·900
·8937	27·633	·9410	14·575
·8967	27·038	·9455	13·250
·8983	26·751	·9510	11·925
·9000	26·500	·9564	10·600
·9015	25·175	·9614	9·275
·9090	23·850	·9662	7·950
·9133	22·525	·9716	6·625
·9177	21·200	·9768	5·500
·9227	19·875	·9828	3·975
·9275	18·550	·9887	2·650
·9320	17·225	·9945	1·325

The gravity may be taken by an ammoniometer, or spirit hydrometer, or in a specific gravity bottle, which, at the ordinary temperature and pressure, holds 1000 grains of water.

Prepared by the usual method, ammonia is liable to be impregnated with chloride of ammonium and ammonium sulphate, which are sublimed, and carried over mechanically. They may be detected by neutralizing a portion of the alkaline solution with pure nitric acid, and testing the liquid, divided into two portions—one with barium chloride, for sulphuric acid; and the other with silver nitrate, for hydrochloric acid. A white precipitate, or milkiness, will in either case prove the presence of the acid sought. Should any lime or calcium chloride be carried over, which is rarely the case if ordinary precautions have been taken, it may be detected by evaporating the solution to dryness, and heating the residue to dull redness for a short time, to expel any traces of ammoniacal salts that may be present; the impurities will remain. If volatile organic substances be mixed with the compounds from which the ammonia is derived, they may be carried over with the vapour and condensed in the receiver; such matters are detected by the brownish or dark colour they communicate to the solution. These impurities may, for the most part, be removed by filtering through animal charcoal. The charcoal absorbs some of the ammonia, and should be washed occasionally with water to abstract it; the washings may afterwards be distilled.

CHLORIDE OF AMMONIUM.—*Sal-ammoniac, ammonium chloride, muriate of ammonia. Chlorure d'ammonium*, or *chlorure ammoniaque*, French; *chlorammonium, salmiak, salzsaures ammoniak*, German. Formula, NH_4Cl.—The manufacture of chloride of ammonium was first practised by the Arabians, although it has been attributed to the Egyptians. The term *sal-ammoniacus* is very ancient; but whether the ancients, at the period in which this term was used, applied it to our chloride of ammonium is doubtful. PLINY and his contemporaries were unacquainted with sublimation in any shape; and it is a fact, that even the process of lixiviating earths, or crystallizing saline substances from their solutions, was so ill understood that the native compounds, however impure, were employed in the manufacture of inks, colours, &c. From the many evidences adduced by BECKMANN, it is almost certain that the sal-ammoniacus of the ancients was common *rock salt*. Indeed, the uses to which it is stated the sal-ammoniacus was applied, leaves no doubt that the chloride of ammonium was not meant. The Arabian philosophers were the first to furnish an account of true sal-ammoniac. GEBER describes its preparation and its purification by sublimation.

Sal-ammoniac was an article of trade about the year 1410; but the purposes to which it was applied, or the mode of its preparation in Egypt at that period, have not been recorded. In the sixteenth century many of its properties were known, particularly its behaviour with nitric acid, by which aqua-regia is formed.

The younger GEOFFROY was the first to show that sal-ammoniac consisted of hydrochloric acid and volatile alkali, and that it could therefore be produced in Europe by sublimation. An account of the sal-ammoniac—ammonium chloride—manufactories at Damayer, in the Delta, was given in 1720 by a Jesuit named SICARD. It is uncertain at what period the manufacture became general in Europe. Large manufactories of this article were at work in Scotland so early as 1750.

Chloride of ammonium was discovered in the mineral products of volcanic districts in the fifteenth century. It is generally met with, sublimed among other volatile bodies, in the fissures of lava, particularly at Vesuvius, Etna, in the Island of Volcano, and at the Solfatara, near Naples. Small quantities of it have been found in the vicinity of ignited coal-fields, as at St. Etienne in France, in Scotland, and at Newcastle. A variety, possessing a greyish-white colour, of a conchoidal fracture, has been mentioned as occurring in Bucharia. The native salt is generally massive, has a fibrous texture, is sometimes feathery, in crusts, and in minute cubic and octahedral crystals; its colour, when pure, is white. It is sometimes transparent, sometimes opaque; externally dull or glistening, and internally shining or vitreous. This salt has no smell, but a pungent and saline taste: its specific gravity is 1·5. It is soluble in 2·72 parts of water at 66° Fahr. (18°·8 C.), with great reduction of temperature, and in its own weight of water at 212° Fahr. (100° C.); it does not attract moisture on exposure to the air. Ammonium chloride is, when exposed to the air, partially decomposed, it loses ammonia, and becomes acid to test paper. When heated it completely volatilizes in white fumes without fusing. After sublimation it forms very tough crystalline masses, which can scarcely be powdered, and emits, when triturated with lime, a strong ammoniacal odour.

Two samples, from Vesuvius and Bucharia, yielded according to KLAPROTH the following results:—

	Vesuvius.	Bucharia.
Ammonium chloride,	99·5	97·5
Ammonium sulphate,	0·5	2·5
	100·0	100·0

Chloride of ammonium is prepared in the laboratory by neutralizing pure ammonia with hydrochloric acid; or by transmitting a stream of ammonia gas into hydrochloric acid till saturated, and then evaporating the solution and crystallizing out the salt. The properties of the chloride, in a chemical point of view, are not very distinctive, though in a manufacturing light the compound is highly important. Its chief characteristics will be mentioned after the reader becomes familiar with the chief methods followed in its preparation on a large scale.

In Egypt, which undoubtedly was the great seat of its manufacture from the thirteenth to the middle of the seventeenth century, and from whence all the European markets were supplied, it was obtained by the following process, which indeed is still in use.

The great source of sal-ammoniac in Egypt is the dung of the camel, which is dried by plastering it upon the walls, and then used for fuel, which is very scarce in that country. A fire of this material evolves a thick smoke charged with chloride of ammonia, a large part of which is condensed in the soot. The latter is carefully collected throughout the country, particularly in the Delta district, and brought to the sal-ammoniac factories for the purpose of distillation. The first accurate account of the manner of manufacturing the salt in Egypt was transmitted thence to Europe by LEMÈRE, French consul at Cairo, in 1770. The soot was put into large round glass bottles, which were externally coated with loam, in order that they might the better bear the heat, and were then ranged in a long ridge or terrace, in such a way that only their necks were exposed to the air, while the flue from the furnace circulated freely round each of them. These bottles, or globes, were generally 1½ foot in diameter, and the neck about 2 inches long: 40 lbs. of soot formed the charge, filling each of them to within 4 inches of the neck. From this weight of material about 6 lbs. of the salt were obtained. A fire, made of dry camel's dung, heated the range of bottles; on the second day, when the bottles became heated, the salt commenced subliming, and much care then became necessary on the part of the attendant to keep their necks free for the passage of the uncondensed vapours. Towards the conclusion of the operation, the fire was urged to bring the globes to incipient redness, that the last portions of the salt might be disengaged. At the end of the third day the fire was extinguished, and as soon as the globes cooled they were taken out, broken, and the cake of salt abstracted, and in this form was exported to Europe. On breaking the bottles, a nucleus of the salt was found surrounded by a fixed pulverulent substance, which was taken out and mixed with fresh portions of the soot in the succeeding operation. Considerable quantities of the salt were evidently lost by this mode of preparation.

In India ammonium chloride was manufactured in much larger quantities than in Egypt. The cakes from the subliming vessels had the form of a sugar loaf, and weighed about 15 lbs. These loaves were composed of three distinct layers; the upper of each was impure, and was generally cut off. Egyptian and Indian sal-ammoniac was a dull, spongy, and greyish mass, considerably inferior to the sal-ammoniac now made; nevertheless, on account of its scarcity, it commanded a price more than six times that of pure ammonium chloride at the present time.

Before the introduction of gas-lighting, the ammonium chloride made in Europe was obtained from offal, or from the refuse cake of oil presses, either by allowing the ammoniacal compound to undergo putrefactive fermentation, or by submitting it at once to destructive distillation; the latter course was adopted in Germany. In this country the soot deposited from pit coal was at one time employed to yield the salt. The distillation of pit coal affords considerable quantities.

At present ammonium chloride is extensively produced as a bye product in the manufacture of bone charcoal. The bones are distilled in iron retorts. The accompanying engravings may be taken as typical of the arrangements commonly made. Figs. 8, 9, and 10 represent a furnace for distilling bones, and obtaining the ammoniacal compounds and the oil from them.

Fig. 8.

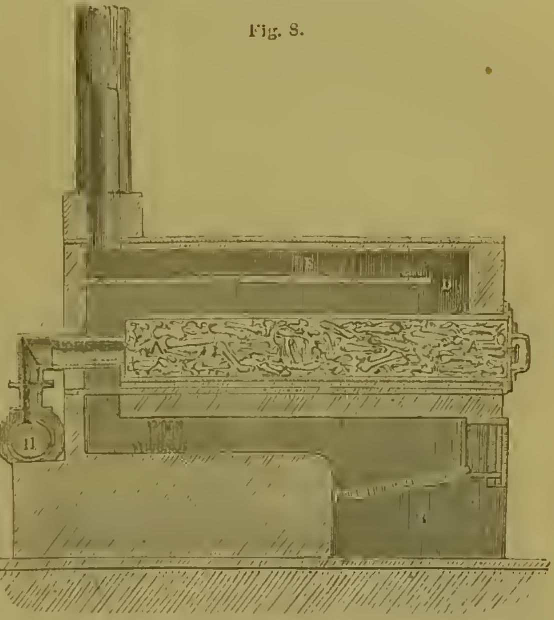


Fig. 8 is a longitudinal vertical section of the retort, A A, for holding the bones, showing its position in the dome of brick which encloses the fire, B. The flue, after passing round the retort, is shown to advance to the front of the furnace, as at D, before it returns by the arch, E, and enters the chimney, J, at the back. The pipe, G, emerging from the far end of the retort, enters the cylinder, H, Fig. 10, into which it conducts all the eliminated products of the distillation.

Fig. 9.

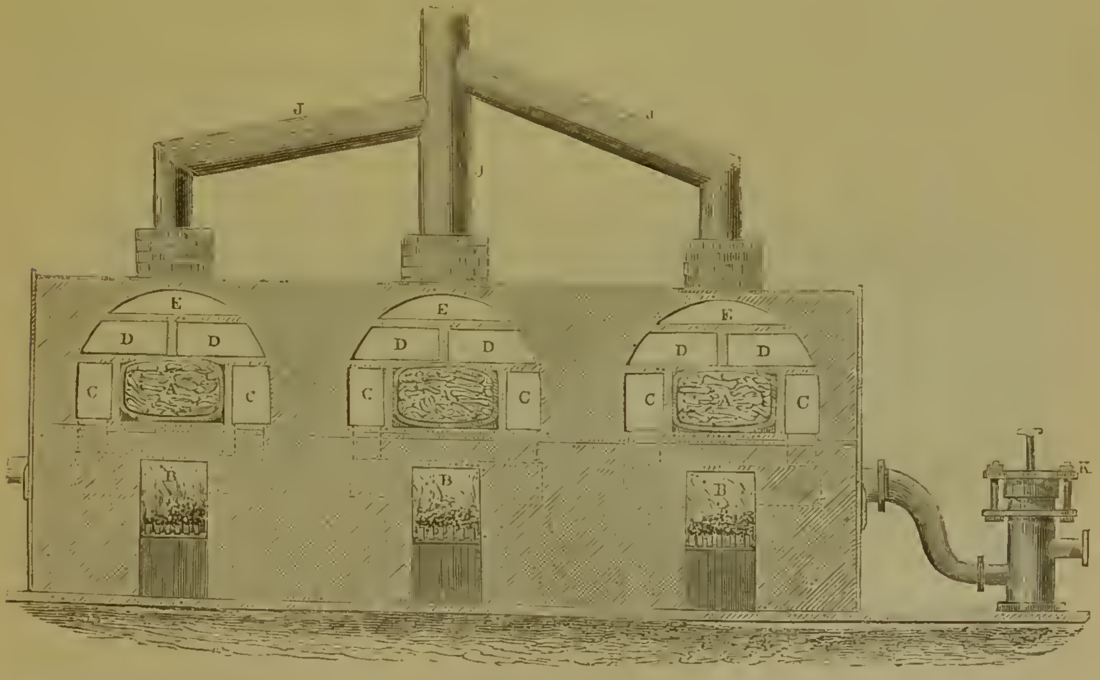


Fig. 9 is a vertical section of the series of retorts and furnaces, made at right angles with Fig. 8, and parallel to the front elevation. A A A are the retorts, and B B B the furnaces, placed immediately under, though not in contact with them. This figure shows more clearly the circuitous passage of the flame and heated strata of vapour from the fires, B, surrounding the retorts laterally at C C, and horizontally at D D, above, as well as at the fireplace below. When the heated air reaches the front of the chambers, D, it returns, as seen in Fig. 8, by the space, E, to the chimney, J. On the right is shown the safety-valve, K, by which air is admitted into the apparatus when the distillation of a charge has been finished; and at the opposite side is seen a portion of the pipe which conducts the uncondensed vapours to the refrigerator.

Fig 10.

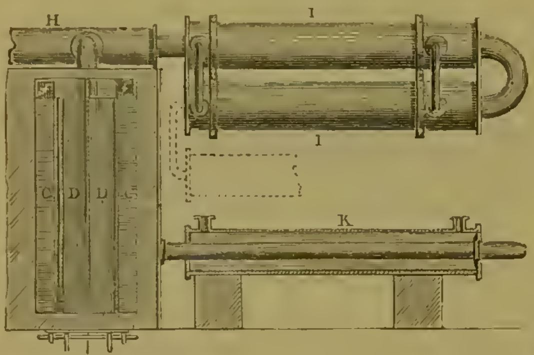


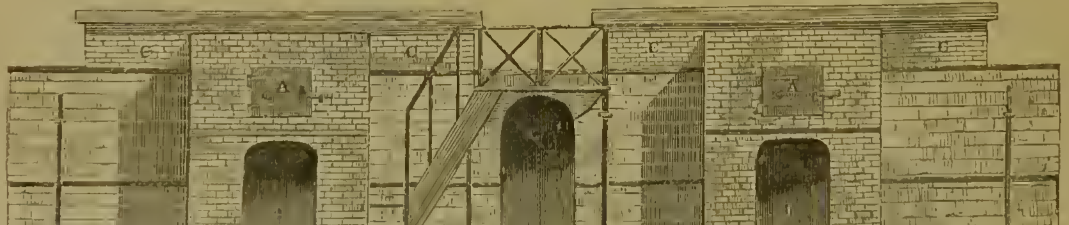
Fig. 10 exhibits a horizontal section of the flues surrounding the retorts, with a view of the condensing pipes from above. In this figure the flues are seen to rise from the canals, C C, to the chambers, D D, at the back, through *f f*, and from D they pass off similarly to E, at the front, as in Fig. 8. The condensing pipes, H and I I, are seen vertically; the latter are rather inclined, and enclose the entrance pipe, while the surrounding space is kept replenished with cold water, which enters at the lower, and is discharged at the upper pipe as it becomes warm. This arrangement is shown in the section, K. From the end of the last refrigerator, the condensed products are discharged into a suitable receiver, where the oil collects at the top in a layer, and is skimmed off, leaving the ammoniacal liquor, to which hydrochloric or sulphuric acid is added in proper proportion till the solution is neutralized; the liquid is then pumped, or otherwise transferred, to the evaporating pans to be concentrated.

Fig. 11 represents a range of such evaporating pans. A A are the fires, B B the ash-pits, and C C C C the evaporating pans, which are about 9 feet square, and 18 inches in depth, and are formed of sheet-lead; their front rests upon a dome of fire-brick, and the remainder is supported by cast-iron plates, resting upon supports of brickwork, which serve to disseminate the heat of the furnace under the bottom of the pan.

In Fig. 12, A shows the fire-bars of the furnace, and C the course of the flame, interrupted repeatedly by the upright brick walls, serving the double purpose of equalizing the heat under the pan, and of supporting the iron plates on which it is laid.

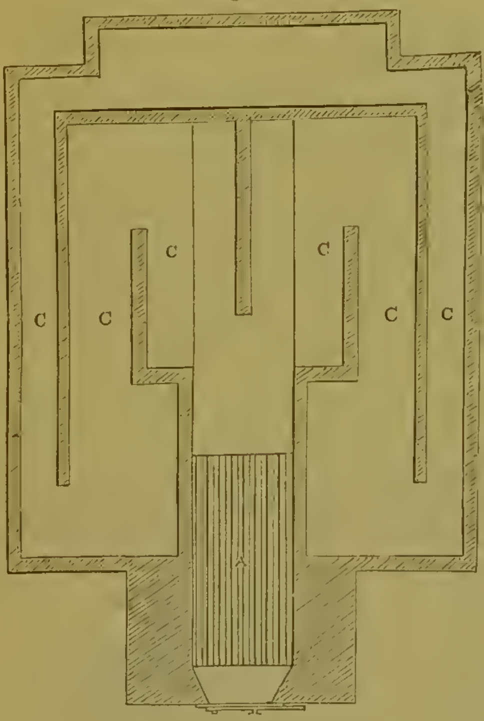
The impurities which pass off in large quantities from the distillation of the bones are condensed in the liquid, and are almost entirely separated in the tank on the addition of the acids. Before evaporation, it is found necessary to remove them completely, which is most conveniently done by a process of filtration. The filters consist of a long wooden box lined with sheet-lead, and having an outlet pipe at its lowest end, for the purpose of drawing off the liquid into the receiver. A frame, corresponding to the shape of the vessel, is adjusted by means of wedges, perfectly horizontal, and within 1 inch of its bottom. Round bars of wood are fixed lengthways in this frame, and upon it a canvass or stout linen cloth is drawn tightly, being secured at the sides and ends of the frame by means of tacks. On passing the liquid through this cloth, most of the solid impurities are retained; and in order that there may be as few of these as possible in the liquid, it is returned by means of a pump from the tank two or three times, to undergo fresh filtrations. A common receiver, placed below the others, may be made to communicate with any of the filters by means of intermediate stopcocks.

Fig. 11.



When the liquid is sufficiently clear, it is pumped up into the evaporating pans just described, and concentrated. As soon as the liquid acquires a certain gravity, it is drawn off to the crystallizing pans. These are wooden boxes lined with sheet-lead, and are of various dimensions, from 6 to 10 feet in length, 3 to 6 feet in breadth, and 1½ to 3 in depth. When the mass of salt is crystallized, the crystallizers are inclined towards either side, in order to drain off all the mother liquor, which is received in a suitable cistern, and is afterwards pumped into the evaporating pans, to be concentrated with another portion of the ammoniacal liquor; the pump employed is made of lead, hardened with antimony and tin.

Fig. 12.



When the crystallized chloride of ammonium is well drained, it is submitted to the subliming process. The arrangement used for this purpose is represented in the annexed woodcuts, Figs. 13 and 14.

The sublimers are earthen or stoneware pots, placed in two ranks, upon the arch of the flue, passing from the fire, A, which heats them. The longitudinal section of the furnace—Fig. 13—shows one row of these pots. Under the pots, represented in the engraving by D, circular openings from the main flue are made in the arch of brickwork, by which the heated vapour and flame are made to circulate around them, and thus they become hot enough to sublime the salt. The bottles, or pots, are encased in the furnace as far as their necks, as seen in the front view and transverse section in Fig. 14; and a plate of iron, with indentations corresponding to the size of the bottles, runs along between the two lines, as shown at B B. The space between this plate and the dome on which the bottles rest is left for the free circulation of the flame and heated air of the furnace, which effects the sublimation. To economize fuel, an evaporating pan is often appended to the subliming furnace; the heated air from the chamber of sublimation is in this case, as it passes to the chimney, made to run along a number of square openings, over which the evaporating pan is supported by cast-iron plates, and at the end of this pan they unite into one flue, just before entering the chimney.

The salt is generally dried in this manner previous to being sublimed.

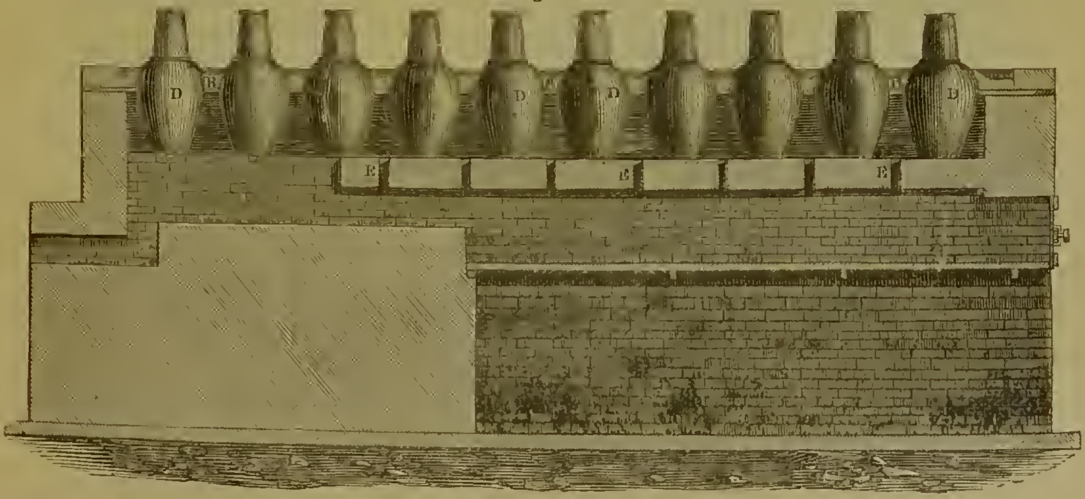
The pots are of variable dimensions, but those most frequently employed are about 18 inches in height in the body, the caps being about 10 or 12 inches, their breadth is 16 inches at the widest part. The annexed numbers show the produce of a large French manufactory of ammonia and its salts, from the distillation of bones and other matters. The materials were—

46,754	tons of	bones of various kinds,
30	"	silk waste and old leather,
11½	"	sulphuric acid,
80	"	chloride of sodium, and
2¾	"	sulphate of lime;

and the produce—

2400	tons of	animal charcoal,
44	"	chloride of ammonium,
100	"	sulphate of soda,
4	"	liquor ammonia, and
25	"	sulphate of ammonia.

Fig. 13.



In this manufactory the sulphate of ammonia is obtained by treating the ammoniacal liquor—which contains chiefly carbonate of ammonia—with sulphate of lime, when double decomposition takes place, carbonate of lime and sulphate of ammonia being formed. The latter remains in solution, and may be obtained by filtration from the lime precipitate, and subsequent evaporation of the solution in the manner indicated by the preceding sketches of the apparatus employed.

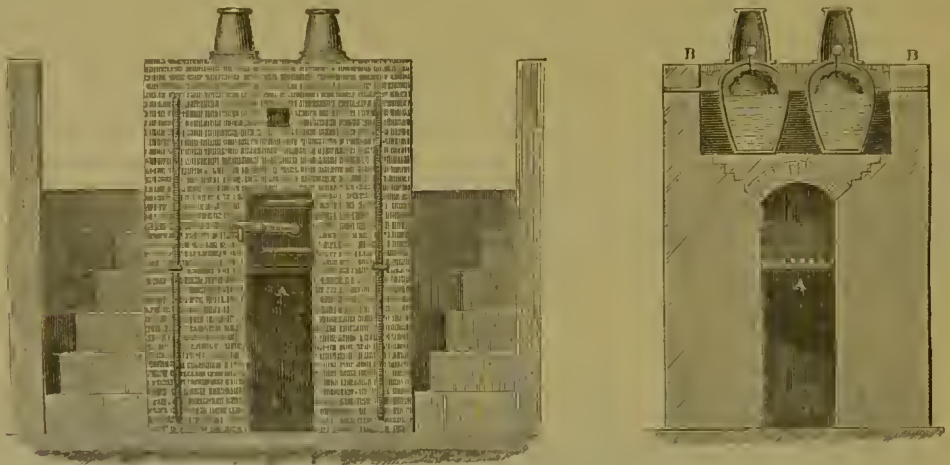
The chloride is prepared either by adding the hydrochloric acid at once to the crude liquor, and evaporating it, or by converting the ammonia into the sulphate, and decomposing this salt by sodium chloride, forming sodium sulphate and ammonium chloride; the latter is separated by sublimation. Occasionally, manganese chloride, from the refuse liquor of chlorine stills, is used to decompose the crude ammonium carbonate; manganese carbonate precipitates, and is separated by filtration, and the solution of the ammonia salt is treated as in the other instances.

When offal is used to produce ammonia by destructive distillation, it should not be heated beyond the temperature required to disengage the ammoniacal compounds, as the residual charcoal can then be made available for the manufacture of prussian blue. According to Ure, the density of the liquor from the distillation of the bones and other substances, when mixed, is 1·060 sp. gr.

When ammonium chloride is prepared by means of sulphate of lime and common salt, the following is the method pursued:—

The crude carbonate of ammonia is decomposed by passing it through a bed of sulphate of lime, 3 or 4 inches thick, which is spread out upon filters. The liquor may be poured upon the gypsum by means of a pump. It should never stand higher than from 1 to 2 inches above the surface of the coarsely ground sulphate of lime, and to prevent the dissipation of any of the ammonia, the vessel should be closely covered over with boards. When the liquor has passed through the first filter, it must be pumped upon the second; or the filters being in a terrace form, the liquor from the first may flow down upon the second, thence upon a third, and so on in succession. The last filter should be nearly fresh gypsum, so as to insure the thorough conversion of the carbonate into sulphate. The resulting layers of carbonate of lime should be washed with a little water, in order to extract the whole of the sulphate of ammonia.

Fig. 14.



The ammoniacal liquor thus obtained must be completely saturated, by adding the requisite quantity of sulphuric acid; even a slight excess of the acid can do no harm. It is then evaporated, and the oil, which is generally carried off in the ammoniacal liquor in combination with the alkali, and is set free by the slight excess of acid, must be skimmed off in the course of the concentration. When the solution of ammonium sulphate has acquired the density of 1·160, sodium chloride is added with constant stirring, until the whole quantity required for the double decomposition is introduced into the boiler. The crude liquor must now be drawn off by a siphon into a somewhat deep reservoir, where the impurities are allowed to subside; it is then evaporated by boiling, till the sodium sulphate falls down in granular crystals, being the result of the mutual action of the ammonium sulphate and sodium chloride, thus:—

$$\overset{\text{Ammonium Sulphate.}}{(NH_4)_2SO_4} + \overset{\text{Sodium Chloride.}}{2NaCl} = \overset{\text{Ammonium Chloride.}}{2NH_4Cl} + \overset{\text{Sodium Sulphate.}}{Na_2SO_4}.$$

while the ammonium chloride, being more soluble, remains in the liquid. During the precipitation of the crystallized sulphate of soda, the whole of the liquid must be agitated by wooden paddles or spatulas, the precipitate being in the intervals removed to the colder part of the pan; thence it is taken by means of copper rakes and shovels, and thrown upon the draining copper placed near the edges of the pan. The drained sodium sulphate must be afterwards washed with a little cold water, to extract all the adhering ammonium chloride. The liquor thus freed from the greater part of the sulphate, when sufficiently concentrated, is to be drawn off by a leaden siphon into the crystallizers, where, at the end of twenty or thirty hours, it affords an abundant crop of crystals of ammonium chloride. The mother liquor may then be run off from the crystallizers, which should be placed in an oblique position to drain the salt; when this is done, the crystals must be washed, first with a weak solution of chloride of ammonium, and next with a small quantity of water. The mass of crystals is then desiccated in a furnace, where all the moisture is removed, after which it is put into the earthen subliming bottles by means of a funnel, and rammed down tightly; the heads are then placed securely on, and heat cautiously applied, so as to effect the sublimation of the pure salt from the bottom to the upper part of the bottle. The vent hole, as shown in the section of the bottles in Fig. 14, must be cleared from time to time, by means of a long steel skewer, to prevent the risk of choking, and consequent bursting; in spite of these precautions, however, several of the bottles are generally cracked in almost every operation.

The preceding method of sublimation is not often adopted in the manufactories of ammoniacal salts in this country. A process somewhat analogous is pursued in Glasgow, where the salt is expelled from cast-iron pots lined with fire-proof tiles; the vapours are received and condensed in a hollow globular head of green glass, with which each of the iron pots is capped. The chloride of ammonium thus formed is sold in hollow spherical masses, corresponding in shape to the head of the vessel, being previously freed from any adhering impurities by mechanical means. The residuary matter left in the pots, which still retains some of the salt, is lixiviated, and the liquor worked up in another operation.

Ammoniacal salts are now more extensively produced from the crude liquor of gas-works than from any other source; this entirely dispenses with the disagreeable necessity of collecting and storing putrid urine for the purpose—a practice formerly extensively followed.

In a large Glasgow factory, where upwards of 70,000 gallons of gas liquor are consumed weekly, it undergoes the following treatment:—The liquor is first rectified by distillation from a waggon-shaped wrought-iron boiler, into a square cistern of iron lined with lead: 4500 lbs. of sulphuric acid, of specific gravity 1·625, are then added slowly to the somewhat concentrated distilled ammoniacal water. The produce is about 2400 gallons of solution of sulphate of ammonia, slightly acidulated, of spec. grav. 1·150, being of such strength as to deposit a few crystals upon the sides of the leaden-lined iron tank in which the saline combination is made. This liquid is decomposed with common salt to obtain the chloride.

The following routine is practised at the factory of Messrs. Kurtz, Cropper, & Co., in Liverpool. The gas-works supplying the crude liquor are situated on each side of the factory, at the distance of three-quarters of a mile; a communication is opened by means of a canal, on the banks of which are the two establishments. Flats, constructed of sheet iron, and divided into compartments of known capacity, convey the ammoniacal liquor to the factory. As soon as the flat comes alongside, the gas liquor is drawn off by a hose, 8 or 10 yards long, to a receiver placed within the walls of the premises; thence it flows through a conduit pipe to large subterranean cisterns, capable of holding 100,000 gallons or more, which are situated nearly in the middle of the factory, and adjacent to large tuns, each capable of holding 14 to 18,000 gallons. Fig. 15 shows the front of these tuns, and the general apparatus appended to them.

By means of a pump, A, connected with the reservoirs, the crude liquor is raised into the tuns; and after a considerable quantity has been pumped, strong hydrochloric acid, in the proportion of 1½ or 2 lbs. to the gallon of the crude liquor, is introduced by the aid of a pulley, or crane, and gutta percha carboys, as shown in the engraving. Metallic pumps cannot be used, on account of the corrosive action of the acid, and even pumps made of gutta percha do not answer.

When the proper quantity of acid has been added, both liquors are intimately mixed by an agitator placed within the tuns, and worked by the same machinery as the pump in connection with the subterranean reservoir; this machinery, however, is not seen in the figure. Disagreeable vapours, consisting of sulphuretted hydrogen and other injurious gases, are disengaged in large quantity during the saturation of the liquid; these are not permitted to escape into the atmosphere, but are made to traverse the fire belonging to the steam engine, being conducted thither by the pipes, D D.

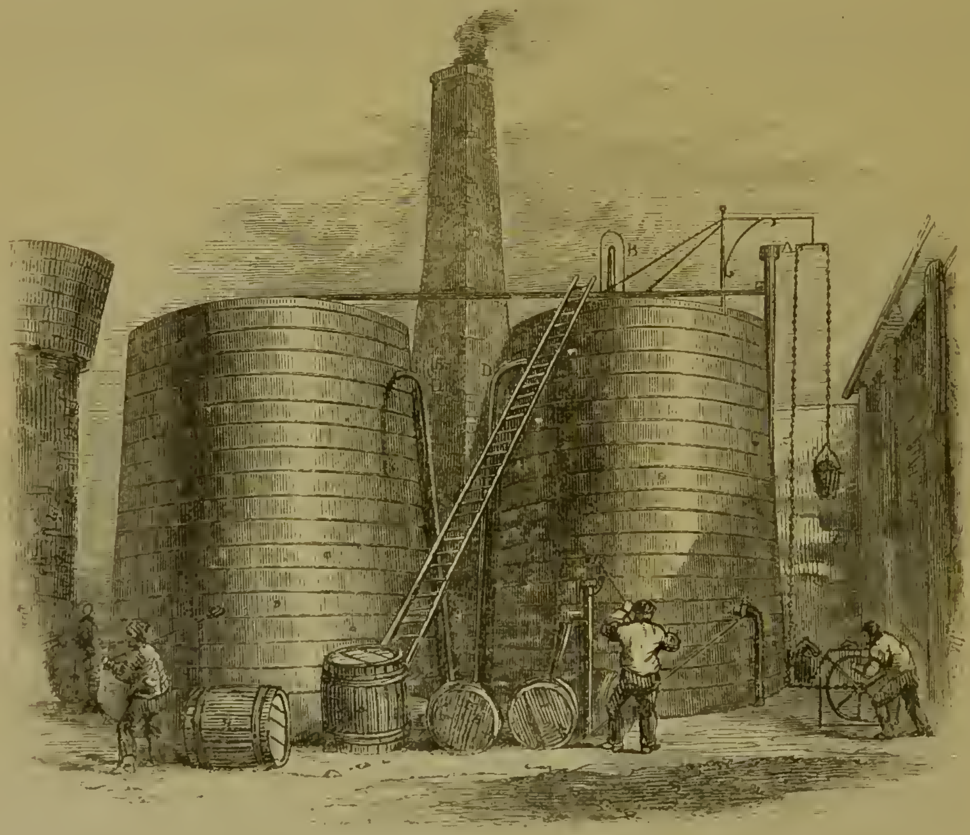
The liquor in the tun should have a faint but distinct acid reaction. The hydrochloric acid, besides uniting with the ammonia, causes the tar and other bodies, held mechanically and in solution, to separate and subside to the bottom of the tun in the course of three or four days, leaving the supernatant liquor much purer, but still deeply coloured.

The liquor is drawn off at several outlets, shown by the dots in the sides of the tuns, but which are kept plugged during the previous part of the operation, and conducted by small sluices or troughs to the store vats and the several concentrating pans, all of which are constructed of iron plates riveted together. The deposits of tar and other matters are drawn off at the lower openings, or by the tap in the pipe, E, when they have accumulated to that elevation (though this rarely happens), to a reservoir adjoining, whence they are pumped into barrels, to be carried to another department of the factory. When there is a superabundance of crude ammoniacal liquor, it is conducted by the pipe, E, to the sulphate of ammonia works of the establishment.

The evaporators are square, rectangular, or circular iron vats, capable of holding from 800 to 1500 gallons; some of them are partly incased in brickwork. Heat is usually applied by a fire, the flue of which takes a sinuous course beneath the lining of brickwork on which the pan rests. During the concentration considerable quantities of petroleum and other impurities, not deposited in the tun, separate, and are immediately removed by skimming. The liquor, which marked only 5° Twaddle previous to being submitted to the action of heat in the pans, is of 50° Twaddle, or 1·25 specific gravity, when transferred to the crystallizers. The solution is slightly acid when drawn over from the mixing tun, and this acidity becomes more distinct as the excess of water is dissipated, and might be productive of inconvenience if it were not neutralized from time to time by the addition of quicklime or chalk in small quantities, or by saturating it with ammoniacal liquor. The neutralization also precipitates any sesquioxide of iron present, and which might prove mischievous in subsequent operations. The liquid being brought to the gravity 1·25, as above noted, is run off to the crystallizers, which are for the most part circular tubs, from 7 to 8 feet in diameter, and 2 to 3 in depth; they are partly imbedded in, or rest upon, the ground.

Fig. 16 is a view of a range of these vessels; they are so placed that the concentrated solution of the chloride of ammonium can be drawn into them from the boilers.

Fig. 15.



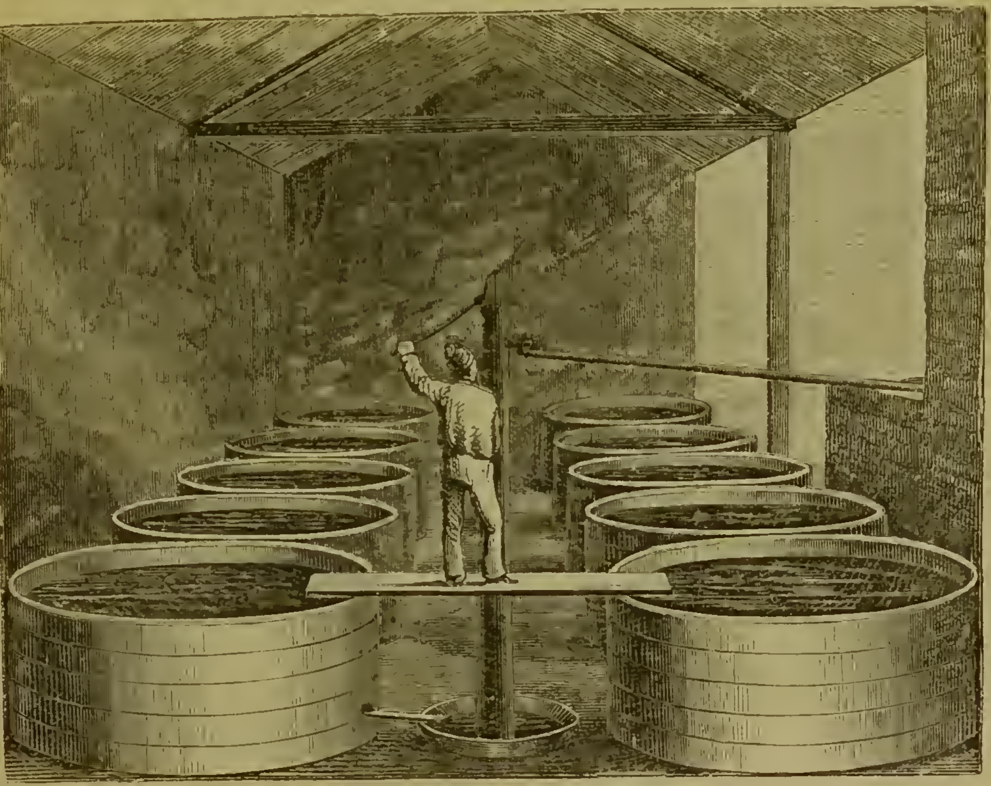
The salt crystallizes out in from four or six days, according to the state of the weather. In crystallizing ammonium chloride, it is not desirable to have large and well-defined crystals, especially when the salt is to be afterwards sublimed; since the process is much more difficult with such crystals, on account of their cohesiveness, than with the more divided salt. On the contrary, when small crystals are submitted to the heat, the cohesion offers scarcely any resistance, a greater surface being exposed, so that the sublimation proceeds rapidly. Hence, when the solution of the salt is set to solidify, the crystals which form on the top, as well as those on the sides and bottom of the vessels, are broken by agitating the liquor every six or eight hours, according to its state of concentration. Eight or ten days are allowed for the salt to crystallize, after which the mother liquor is removed through an opening at the bottom, to a well sunk in the ground, whence it is again pumped into the concentrating pans as shown in the engraving.

Ammonium chloride, as obtained by the first crystallization, is a blackish salt, interspersed with moderately large cubical crystals. The blackness arises from tarry, oleaginous, and other impurities, mechanically held in the liquor, which fall down, more or less, with the crystals; the chloride also contains sulphates and hyposulphites and water, these all tend to deteriorate the quality of the ammonium chloride, if not removed before sublimation. To get rid of the impurities, the well-drained crystals are introduced into the bed of a drying furnace, covered with a cast-iron plate, the whole surmounted with a dome of brickwork, and erected near the sublimers. This drying bed is heated by a fire placed under it, the flue of which is brought several times into such a position that the plate is within its influence. This chamber is between 6 and 8 feet in length, and about 4 feet in breadth, and a sufficient charge is introduced to cover the entire plate to the depth of 4 inches. The heat should never be raised very high, lest some of the salt be volatilized. When all the water and free acids are removed, and the empyreumatic impurities for the most part decomposed, the greyish-white mass is drawn out and taken to the subliming apparatus, where the salt is eliminated from the non-volatile impurities.

The subliners are circular spaces, constructed in a mound of brickwork, walled interiorly with fire-brick, and lined with cast-iron plates, round the sides of which the flue from the fire underneath coils, after it passes from the bottom. The subliming vessels are capped by an iron cover, somewhat of the shape of a flat-bottomed watch-glass. They are from 3 to 9 feet in diameter, and their concavities about 2; they weigh from 10 to 30 cwts. Three rings, at equal distances, allow of their being lifted off by means of a pulley and chains which can be attached to these rings. When in active operation a pressure of from 3 to 4 lbs. per square inch bears against them, which requires their being firmly fixed to the flat rim of the body of the subliming vessel. In the centre of some of these is a small hole, closed by an iron spindle, which is removed from time to time, to allow any elastic uncondensable products to escape.

The annexed woodcut, Fig. 17, is a view of the sublimers and drying furnaces. They are heated by the fires, which are briskly kept up till the sublimers and their surroundings attain a sufficient degree of heat; they are then slackened, and maintained at an even temperature till the operation is completed.

Fig. 16.



Much of the success of the sublimation depends upon the regulation of the heat. If the temperature is too high large quantities of empyreumatic bodies are disengaged, which interfere with the operation by preventing the solidification of the salt; and should the temperature be too low, there will be a looseness of texture and an opacity in the salt which greatly deteriorates its value in the market. The top of the sublimer is invariably covered with some non-conducting material, that the heat may be economized as much as possible.

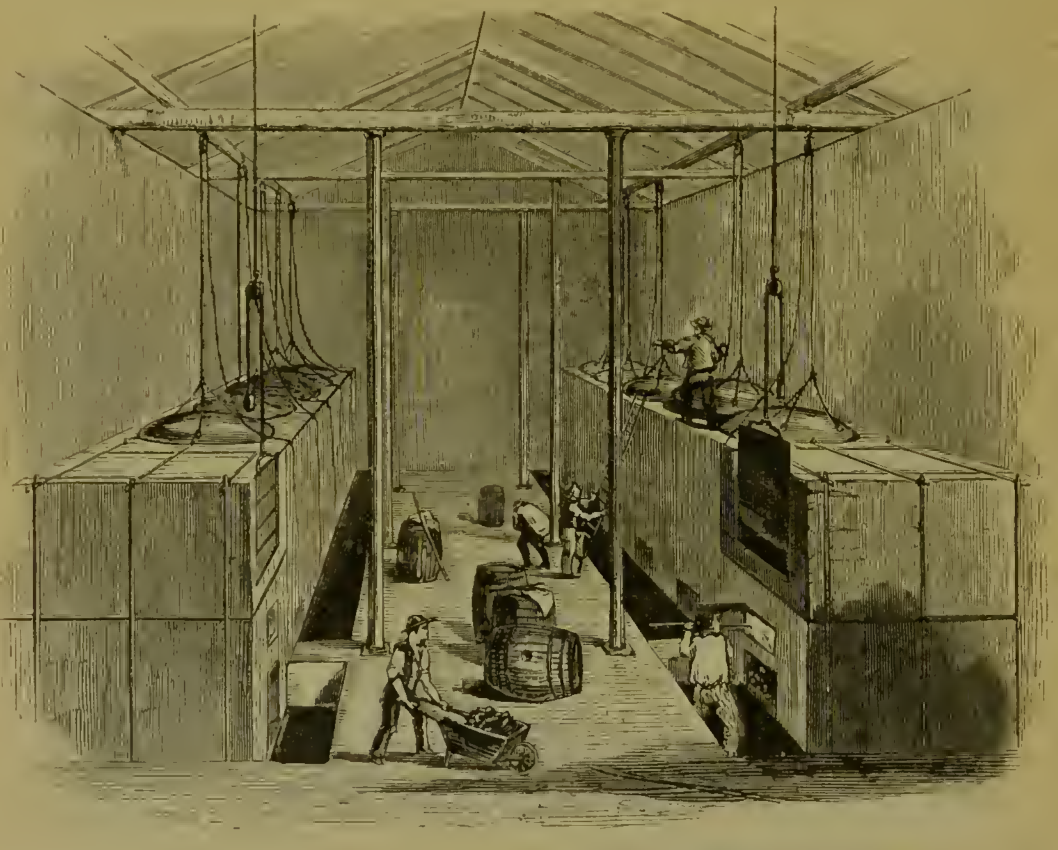
As the size of the sublimers varies, as above stated, so also does the amount of the charges, being from ½ to 2 or 2½ tons weight of the salt; the former is sufficient for sublimers of 3 feet diameter, and the latter for those which are 9 feet; sublimers of 5 feet diameter generally require from 15 to 18 cwts. of the salt.

In consequence of ammonium chloride retaining some traces of water, even after passing through the drying furnace, and as it always absorbs a little from the atmosphere while the workmen are transferring it to the sublimers, the first coating of the salt in contact with the cover is always brownish. Several reasons may be assigned for this effect: firstly, the moisture condensing upon the head, and softening any small quantity of ferric oxide present, would cause it to sink into the layer of salt that deposits thereon; secondly, decomposition of any resinous matters not completely destroyed in the drying furnace, whereby oily vapours would be liberated; and lastly, the accidental presence of ferrous or ferric chloride. The effect of the latter, however, is not generally on the first layer, but more frequently afterwards, when the temperature is higher.

The termination of the sublimation takes place in from five to nine days; but it is customary to raise the caps every week, the fire being checked for some hours previously. The salts are not sublimed to their last dregs, because the temperature required would cause the decomposition of the carbonaceous impurities, and emit vapours which would destroy in a great measure the beauty of the sal-ammoniac. When a low temperature is used, or the compound consigned to the subliming furnace is impure, the chloride does not assume that compactness of grain which conduces to its transparency, but forms a kind of effloresced mass, though retaining the fibrous construction of the better quality.

When the sublimation has reached as far as is desirable, the fire is allowed to go out that the apparatus may cool; the cover is then removed by means of the tackle before noted, and there is found in the interior a hard lining of the salt, from 2½ to 3 or 4 inches thick; this is detached and conveyed to the packing-house or store, and the residuary matter remaining in the body of the sublimers is removed to another part of the factory to be converted into sulphate of ammonia. The brown coating, arising from the causes before alluded to, is separated from the cake by means of axes. The greater part of the chloride of ammonium manufactured is employed by colour-makers and calico-printers, and by makers of galvanized iron.

Fig. 17.



SULPHATE OF AMMONIA.—*Ammonium sulphate, sal-ammoniacum secretum*, Glauber; $(NH_4)_2SO_4$.—This salt forms crystals resembling those of potassium sulphate, with which it is isomorphous. They belong to the trimetric, *i.e.*, the right prismatic system. The prisms have six faces, and terminate in six-faced pyramids, belonging to the orthorhombic type.

Ammonium sulphate is colourless, has a sharp bitter taste, and is soluble in its own weight of boiling water, or twice its weight of cold water. In alcohol it is wholly insoluble. When heated it decrepitates, and at 284° Fahr. (140° C.) melts. Above 536° Fahr. (280° C.), it is decomposed into ammonia, nitrogen, water, and acid ammonium sulphite, which latter sublimes. When a solution of ammonium sulphate is subjected to protracted boiling, it is in like manner split up. At a dull red heat it is completely decomposed, and yields sulphur, nitrogen, and water thus:—

$$\underset{\text{Ammonium sulphate.}}{(NH_4)_2SO_4} = \underset{\text{Sulphur.}}{S} + \underset{\text{Nitrogen.}}{2N} + \underset{\text{Water.}}{4H_2O}.$$

Ammonium sulphate is found native (Mascagnite), usually in mealy crusts and stalactitic forms. It collects about the solfatara of Naples, round volcanoes, and in the fissures of the lava, as at Etna, Vesuvius, and the Lipari Isles. This mineral is named after its discoverer, Professor Mascagni.

Manufacture.—Sulphate of ammonia is obtained from gas liquor by the following process:—A large pipe conveys a quantity of gas liquor to a large boiler placed over a fire, the flue from which circulates round the bottom and sides; a large pipe issues from its upper surface top, and descends through an adjoining tun, containing sulphuric acid, to its bottom. By bringing the contents of the boiler to ebullition, the free ammonia is driven off through the pipe into the tun containing acid; here it is absorbed by the sulphuric acid, giving rise to ammonium sulphate. Distillation is continued till no more ammoniacal vapours are given off, and then the fire is checked, while the residual liquor is drawn off and mixed with sufficient quicklime to give it a strong alkaline reaction; the whole is then run into a second and larger boiler, like the preceding, on the principle of a still; under this a brisk fire is applied, and the ammoniacal vapours liberated by the action of the lime are transmitted through the sulphuric acid solution in the tun. After all the ammonia is obtained, the waste liquor is run off.

During the time the ammonia vapour is blown through the sulphuric acid liquor, much water is removed from the solution by the heat from the steam and ammoniacal gas, so that less fuel is required in further concentrating the liquor. The evaporating pans are similar to those used for boiling down the chloride of ammonium solution. Much more care is requisite when fire is applied directly to the pans, than when the concentration is effected at a steam heat; for the organic matter which the liquor contains partially decomposes the sulphate into sulphide, which reacts upon the iron pans, or by the excess of acid present is decomposed, giving off sulphurous acid or sulphuretted hydrogen gas. The operative watches carefully for any traces of the odour resembling that of rotten eggs, which characterizes sulphide of hydrogen, because this tells him that decomposition is taking place, and of course admonishes him that it should be prevented by immediately withdrawing or slacking the fire.

Crystallized sulphate of ammonia obtained in the first operation is much purer than the chloride of ammonium already described, in consequence of the impurities of the crude liquor being removed by the distillation. After the crystallization, the crude liquor which is still acid is returned to the tun, to combine with further quantities of ammonia.

Besides the ordinary gas liquor, the impure chloride from the packing-house and the refuse of the sublimers are decomposed by lime, and the eliminated ammonia either transmitted through the acid solution to form sulphate, or used to prepare the carbonate. The sublimation of the crystallized sulphate cannot well be effected; but ammonium chloride may be prepared from it, by mixing it with a proportionate quantity of chloride of sodium, and then heating; the chloride is sublimed, and sulphate of soda remains in the bottom of the sublimer.

In subliming ammoniacal salts, considerable advantage results from the use of large vessels, as the labour of attending them is less in proportion, and the fire which is requisite to heat a pan of 3 feet in diameter would serve, with very little addition, to sublime the contents of one of 9 feet; the time required also in working the charge of each sublimer, whether it be 3, 5, or 9 feet diameter, is nearly the same, though the quantity of salt produced by the larger ones is very different. A great advantage in speed of working is gained by making the bottom rise in the centre instead of being flat. The heat is thus sooner communicated to the mass of the material, and the time required for sublimation is much shortened. Ammoniacal gas liquor yields from $\frac{3}{4}$ lb to $1\frac{1}{4}$ lb. of sal-ammoniac per gallon of liquor, but this quantity is subject to much variation from many causes.

In the manufacture of ammonium chloride as already described, if the purification of the liquor be not effected before crystallizing the salt, some traces of ferrous chloride are generally present, sometimes in large proportions. When the crystals are submitted to heat in sublimation, the ferrous chloride is volatilized, and blends with the ammoniacal salt in the colourless as well as in the dark or brown seams of the cake.

Wurtz, who examined this substance, states that neither the brown nor colourless portions of the sublimate give any indications of iron with the usual reagents, until nitric acid has been added to convert the ferrous salt into the ferric modification, after which the addition of potassium ferrocyanide produces a very distinct precipitate of Prussian blue, and potassium sulphocyanide a red coloration. It was not generally considered that the colourless chloride of ammonium contained iron till Wurtz discovered this reaction. It seems that the iron might be present in the lie as ferric chloride, and is subsequently converted into ferrous chloride by the reducing action of ammonium chloride. The reaction may be represented as follows:—

$$\overset{\text{Ferric chloride.}}{Fe_2Cl_6} + \overset{\text{Ammonium chloride.}}{2NH_4Cl} = \overset{\text{Ferrous chloride.}}{2FeCl_2} + \overset{\text{Hydrochloric acid.}}{4HCl} + \overset{\text{Amidogen.}}{2NH_2};$$

or—

$$\overset{\text{Ferric chloride.}}{3Fe_2Cl_6} + \overset{\text{Ammonium chloride.}}{2NH_4Cl} = \overset{\text{Ferrous chloride.}}{6FeCl_2} + \overset{\text{Hydrochloric acid.}}{8HCl} + \overset{\text{Nitrogen.}}{2N}.$$

In either case the ferric chloride is converted into ferrous chloride.

Repeated crystallizations would remove the iron compound, but this would cause a loss of time, fuel, and material. The smallest trace of this salt can be separated at very slight cost by passing a few bubbles of chlorine gas through a concentrated hot solution of the salt in the crude state, which reconverts the ferrous chloride into ferric chloride; this in turn is decomposed by free ammonia, with the precipitation of hydrated ferric oxide of iron and the formation of ammonium chloride. The liquor is kept hot for some time, till the whole of the flocculent brownish matter settles to the bottom, after which it is rapidly filtered and set aside to crystallize. By this means a perfectly pure salt, free from the least trace of iron, is obtained. Great care should be taken to keep the solution hot during the time the chlorine is being transmitted through it, and also not to prolong the action, in order to prevent the formation of the well-known dangerously explosive mixture, nitrogen chloride, NCl_3. (This substance is now thought to contain hydrogen, and to be represented by the formula $NHCl_2$ or NH_2Cl).

Croll gives the following instructions for the preparation of ammoniacal salts:—A vessel, similar to those used for purifying coal gas by means of moistened quicklime, is filled with a solution of manganese chloride, made by dissolving 1 cwt. of that salt in 40 gallons of water, and the gaseous ammonia, generated either by addition of lime to gas liquor and heating, or by driving high pressure steam into the gas liquor to which milk of lime has previously been added, is passed through it by the pressure from the retort. The manganic solution absorbs the ammonia, which precipitates a corresponding proportion of oxide of manganese, and as soon as fully saturated it is drawn off, and a fresh charge introduced.

When sulphuric acid is to be combined with the alkali, a vessel similar to that for washing the gas is used, the sulphuric acid used being of specific gravity 1·845, and in the proportion of 2½ lbs. of acid to 100 gallons of water. The gas is passed into it till the liquid marks a specific gravity of 1·170, when it is drawn off and a second charge supplied.

In preparing chloride of ammonium the hydrochloric acid is taken at a density of 1·165 before diluting it, and then charged with the ammonia gas till the gravity is 1·176; the proportion is the same as with sulphuric acid. In the preceding case of the manganese salt being used, the ammoniacal compounds are purified by filtering off the solution from the precipitated oxide, evaporating the filtrate, and subliming the residue. Sulphate of manganese, or ferrous chloride, may be substituted for the chloride of manganese. That portion of metallic oxide which remains is again converted into a fit state to absorb fresh quantities of the alkaline vapour, by the following simple method:—To every three parts of this residue four of common salt are added, and the mixture heated in a furnace to low redness, which is scarcely perceptible in the dark, for about two hours or longer. Forty gallons of water are then added to every 140 lbs. of this mixture, and the liquid thus produced absorbs ammonia very rapidly. The precipitate which falls, consequent on the retention of the ammonia, only requires to be dissolved in the acid which is intended to form a combination with the alkali, to render it suitable to serve the intended purpose a second and a third time.

Hill proposed to convert the ammonia eliminated in the distillation of coals into chloride, by mixing chloride of manganese with the coals in the retort, or introducing the chloride into a retort appropriated for the purpose; the heat dispels the hydrochloric acid, and this, uniting with the alkaline vapour, forms chloride of ammonium, which is retained in the liquor in the condenser After removing the tar in the usual way, the salt is obtained by evaporation and sublimation, as before described. This process was not commercially successful.

Spence uses a series of cylindrical-covered boilers, or reservoirs, placed at such different elevations that the contents of the uppermost may be drawn off into the next, and so on in succession to the lowest, as an economical arrangement in preparing chloride of ammonium, or sulphate of ammonia, from gas liquor, &c. Each boiler has an exit pipe, which carries the vapour generated in it to that which is next above it, and the exit pipe of the highest boiler passes off to a tank, where the acid requisite to form the salt is supplied. A charging pipe connects the top boiler with the reservoir of gas liquor, which is already mixed with milk of lime, and by turning a stopcock attached to it the boiler may be replenished. Four boilers are deemed sufficient, but a larger number may be used. A pipe conveying high-pressure steam enters the lower boiler, by which the liquid is made to evolve its ammonia into the next boiler; and in the same manner the contents of this and the upper boilers are brought to the temperature of ebullition, the ammonia passing through each by the connecting pipes, till it is expelled from the highest, in a very concentrated state, to the acid tank. When moderately concentrated acid is used after the absorption of the ammonia in the series of boilers, the lie is so strong that it may be drawn off at once to crystallize. As the liquor in the lowest boiler becomes exhausted of its ammonia, the fire is slackened and the contents run off, and that of the next boiler admitted instead; the latter is supplied from the one above it, and so on to the highest, which is filled from the reservoir. When the boilers are replenished the fire is stirred up, and the distillation proceeded with as before. The solution of chloride of ammonium, or sulphate of ammonia, is treated in the usual way for obtaining the dry salt.

Salts of ammonia are prepared from guano by destructive distillation in close iron cylinders. A low red heat is maintained during the greater part of the operation; this temperature is increased towards the end. The eliminated gases are made to pass through vessels arranged on the principle of Woulfe's bottles. Carbonate of ammonia, hydrocyanic acid, and marsh gas, are copiously disengaged; the first two, being rapidly absorbed, give a strong solution of ammonium cyanide and carbonate of ammonia, while the marsh gas passes into the atmosphere.

As soon as the charge of the retorts has been exhausted, the solution in the condensing apparatus is drawn off, and mixed with as much ferrous chloride as is necessary to separate the whole of the hydrocyanic acid in the form of Prussian blue. In consequence of the free ammonia present the precipitate does not at once appear on adding the iron salt, but on neutralizing this ammonia with hydrochloric acid it then falls down of a fine blue colour; this is filtered off and well washed, after which it is converted into potassium ferrocyanide (*yellow prussiate*) by boiling with potassa, evaporating the solution, and crystallizing in the usual way. If an excess of iron is present in the liquid filtered from the iron precipitate, it is separated by adding carefully a fresh quantity of the ammoniacal solution, which precipitates it, and it is then removed, and the neutral solution of ammonium chloride evaporated. Sulphate of ammonia is procured in a similar manner, by employing sulphuric instead of hydrochloric acid, and sulphate of iron instead of the chloride.

Peat is rich in ammonia, and therefore the fabrication of ammoniacal salts from this source have formed a main feature in the multitudinous schemes for its utilization.

According to the statements which have been drawn up by SIR ROBERT KANE and others, peat yields from 22 to 25 lbs. of ammoniacal salts per ton.

For the preparation of ammonium chloride or sulphate, LAMING adapted various compounds to be used in the purifiers of gas-works; and through which the gases given off from the retorts are transmitted, so as to retain the ammoniacal vapour. These substances are ferrous chloride decomposed by hydrate of lime into oxide, with the formation of calcium chloride, ferrous sulphate (which he converts by means of chloride of sodium into ferrous chloride), and sodium sulphate of soda. The chloride thus prepared is then decomposed, as in the preceding instance. Further, he uses a mixture of calcium sulphate and ferrous sulphate, or of the precipitated hydrated ferric oxide with calcium carbonate, magnesia, magnesium carbonate, or magnesian limestone; of magnesium chloride or sulphate and water; of phosphate of lime dissolved in hydrochloric acid; and of a mixture containing magnesium sulphate or chloride in combination with oxide of copper, and mixed or not with lime or magnesia, or both or either, or both of the carbonates of those earths. In all these cases the salts or mixtures employed are mingled with sawdust or some porous substance before being placed in the purifying vessel. After they become saturated with ammonia, the chloride or sulphate formed is removed by simply washing the mass with water.

LAMING, later on, employed sulphurous acid to prepare sulphate of ammonia. The ammonia is liberated in the free state or as carbonate, by any simple means, and received in water; by a stream of sulphurous acid gas transmitted through this ammoniacal liquid, it is converted into ammonium sulphite. By agitation and exposure to the air this salt is oxidized, and becomes ammonium sulphate.

Sulphate of ammonia was manufactured by MITCHEL by means of sulphate of lead, containing an excess of oxide. On submitting such compounds to the ammoniacal solutions, decomposition of the lead salt takes place, being converted partly into sulphide, and partly into a carbonate, while ammonium sulphate results. He prepared the oxysulphate in the following manner:—Native sulphide of lead is ground into small fragments; the reduced mass is then spread upon the higher of two shelves constructed in a reverberatory furnace, where it is heated for about two hours. It is then drawn off to the under bed or shelf, where it is more strongly heated, and kept occasionally stirred to prevent fusion or caking of the mass, and also to have every part equally heated. The second heating effects the transformation of a portion of the sulphide into sulphate, while another part is oxidized and sulphur eliminated. The layer of galena is about 2 to 2½ inches in depth.

WILSON obtained sulphate of ammonia from the waste products of coke ovens, &c., by transmitting the vapours through a circular tower filled with coke. A cistern at the base of the tower is filled with sulphuric acid, so much diluted that, when completely saturated with ammonia, the heat of the apparatus will not concentrate it sufficiently to deposit crystals of the salt. Another cistern, similar to the preceding, is placed on the top of the tower, the bottom of which is perforated. All the vapours from the ovens are forced through the lower cistern and up the tower; and by an arrangement which continually raises the dilute acid liquor of the lower tank to the upper, so that it again falls through the perforations, any of the alkali that may escape combination from the cistern is taken up by the trickling solution in the body of the cylinder.

The sulphate of ammonia is obtained from the solution after the acid employed has been neutralized, by filtration, evaporation, and crystallization.

Dr. RICHARDSON proposed to obtain ammonium sulphate by forming a double sulphate of magnesium and ammonium, and then submitting this product to sublimation.

SESQUICARBONATE OF AMMONIA.—*Ammonium sesquicarbonate*, *half acid carbonate*, *tetramonio-dihydric carbonate*, *sal-volatile*, *salt of hartshorn*, &c.; $N_4H_{18}C_3O_9 = (NH_4)_4H_2(CO_3)_3$.—Commercial sesquicarbonate, or carbonate of ammonia, consists of half-acid carbonate more or less mixed with other ammonium salts. This compound is prepared on the large scale by the dry distillation of bones and other animal matter; or by heating ammonium chloride or sulphate together with chalk to redness, in close vessels; double decomposition of the two compounds follows, causing the formation of sesquicarbonate of ammonia, and a lime salt analogous to the ammoniacal one taken.

Leaden chambers are used as condensers to retain the volatilized salt from the heated retorts; they generally have the top or bottom partly movable, or have a door in their sides to allow of the removal of the salt. The crude sesquicarbonate obtained in the first sublimation is purified by expelling it by heat from iron pots surmounted by leaden caps. The waste heat of the flue from the furnace which heats the retorts serves for this part of the work. In the second operation water is added to render the salt translucent.

The annexed woodcut, Fig. 18, represents such an arrangement as is here mentioned. In this figure the retorts are cylindrical, and laid horizontally in the furnace, similar to those used in the carbonization of wood for the production of wood vinegar, having the exit pipe for the passage of the sublimed salt at the back; or they may be like those already described in the distillation of bones.

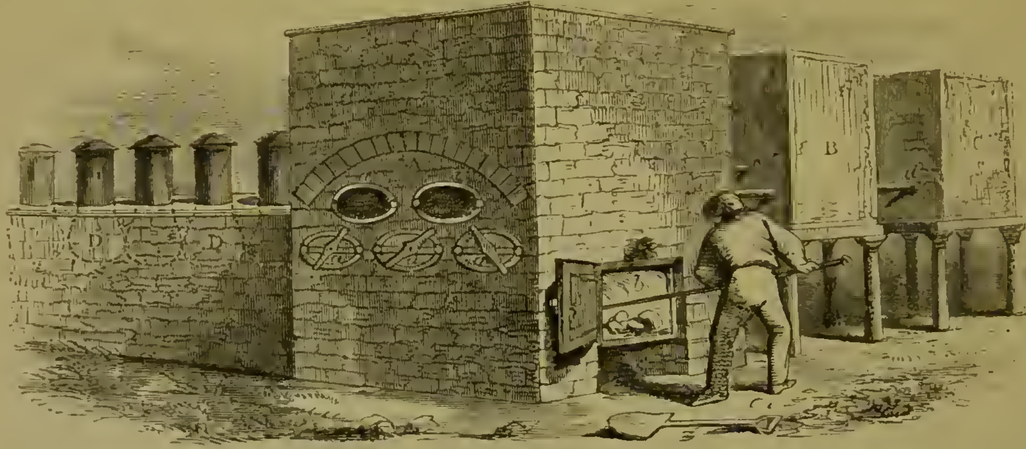
A is the exterior of the furnace containing the five retorts, *a a a a a*, laid horizontally, and closed at the front by iron doors made secure by bolts and screws. Heat is communicated by the fire, *b*, at the end of the furnace. The pipes, *c c*, carry off the vapours of ammonium carbonate into the chamber, B, and whatever remains uncondensed in this is discharged into a second one, C, by the connecting pipe, *d*. Both chambers are supported upon pillars or scaffolding, to bring them on a line with the retorts; D D are the purifying pots, made of cast iron, and surmounted with leaden heads, into which the carbonate is volatilized from any fixed impurities by the heat of the flue from the fire, *b*.

One part of ammonium sulphate or chloride, mixed with 1½ or 2 parts of carbonate of lime, is introduced into the retorts, and a moderate heat applied at first, which is gradually increased towards the end of the operation. The vapours which are eliminated through the pipes, *c c*, in the figure, are condensed in the chambers, B and C. After the charge in the retorts becomes exhausted, the salt condensed in the chambers is removed to the purifying vessels, D D, where it is sublimed at a low degree of heat; sometimes 150° Fahr. (65°·5 C.), communicated by a water-bath, is found to answer the purpose.

Laming recommends, for the preparation of commercial carbonate of ammonia, the conveyance of the constituent gases into a series of large leaden chambers, the temperature of which should be kept as low as practicable, in order to induce as much as possible the combination of those gases. It is unnecessary to have them in combining volumes; in fact, it is desirable that the carbonic acid should be in excess.

A stratum of water, or of a solution of ammonia, is supplied in one or other of the chambers, and in this case the resulting product contains more carbonic acid than when the gases unite as they issue from the vessels where they are generated.

Fig. 18.



The solution is evaporated, and the salt purified by sublimation.

Carbonate of ammonia may be prepared from the ammonium sulphide found in gas liquor by the annexed process. Oxide of copper and charcoal are mixed in the proportion of 12 parts of the former to 1 of the latter; and this mixture is introduced into any suitable retort—cast iron—wherein it is heated to redness; the pipe joining the beak passes through cold water previous to its entrance into the ammoniacal solution of the sulphide or the gas liquor. The action of the heat upon the oxide of copper and charcoal gives rise to carbonic acid, which, entering into the solution, displaces the sulphur in combination with the ammonium, and causes it to be dispersed as sulphuretted hydrogen, while the carbonic acid takes its place, and a solution of carbonate of the alkali is formed.

When the carbonic acid ceases to be given off, the whole of the oxide is found to be reduced to finely-divided metallic copper, and very little, if any, charcoal remains. The retort is cooled, and the contents drawn out, while another retort, similarly charged, maintains the stream of carbonic acid through the solution. During the cooling of the contents of the first retort, the finely-divided copper, by uniting with oxygen from the atmosphere, is reconverted into protoxide, which may be used over again with a fresh portion of charcoal; and so on till the evolution of sulphuretted hydrogen ceases, at which period the liquor is known to be saturated with the carbonic acid gas.

Sesquicarbonate of ammonia is a white fibrous substance, generally found in the market in white translucent cakes of about 2 inches in thickness. It is soluble in 4 parts of water at 55° Fahr., in 3·3 parts at 62°, in 2·7 at 90°, in 2·4 at 105°, and in 2 parts at 120°.

On exposing this solution to moist air, water is absorbed, decomposition takes place, and it is converted into the neutral carbonate and bicarbonate, or acid carbonate of ammonia.

$$\overset{\text{Ammonium sesquicarbonate.}}{(NH_4)_4H_2(CO_3)_3H_2O} = \overset{\text{Ammonium neutral carbonate.}}{(NH_4)_2CO_3H_2O} + \overset{\text{Ammonium bicarbonate.}}{2(NH_4)HCO_3}$$

But the neutral carbonate is very unstable, and speedily resolves itself into the more stable ammonium bicarbonate, at the same time giving off water and ammonia thus:—

$$\overset{\text{Ammonium neutral carbonate.}}{(NH_4)_2CO_3,H_2O} = \overset{\text{Ammonia.}}{NH_3} + \overset{\text{Water.}}{H_2O} + \overset{\text{Ammonium bicarbonate.}}{(NH_4)HCO_3}$$

The same change takes place when solid sesquicarbonate of ammonia is exposed to the air; hence the use of ammonium sesquicarbonate as smelling salts. Bicarbonate of ammonia, remaining after the exposure of the sesquicarbonate, is not so pungent as the salt which produces it. Medically, it is known as mild carbonate of ammonia. It yields a precipitate with bichloride of platinum in the presence of hydrochloric acid; with copper salts it gives the characteristic blue colour produced by ammonia; with the salts of the alkaline earths it does not yield an immediate precipitate; on boiling the mixture, however, the insoluble earthy carbonate falls down.

ROSÉ states that the composition of the sesquicarbonate varies with the mode of preparing it. When obtained by the sublimation of the salt from chloride of ammonium—or any other compound of ammonia—and carbonate of lime, the product is a sesquisalt; but on subsequent sublimation, for the purpose of purifying, he says that it is only four-fifths sesquicarbonate, as his analysis indicates. Sesquicarbonate of ammonia possesses a pungent odour, a hot and saline taste, and a powerful alkaline reaction. The aqueous solution of this salt is used in medicine as a stimulant, and is a useful source of other ammoniacal salts. Bicarbonate of ammonia crystallizes from the hot concentrated solutions of the sesquicarbonate, or it may be precipitated by alcohol. By the action of heat the sesquicarbonate decomposes into various ammonium compounds, containing more or less carbonic acid; the sesquicarbonate of commerce is a mixture of several of these.

Pure ammonium sesquicarbonate is obtained by dissolving the commercial salt in strong ammonia, at about 86° Fahr. (30° C.), and allowing the solution to crystallize. It then forms transparent right-rectangular prisms, with the faces of the corresponding rhombic octahedron resting on the angles. The impurities of the commercial sesquicarbonate are organic matter and small portions of the salt from which it is prepared, which pass over to the chambers unchanged; sometimes it contains hyposulphite of ammonia from the decomposition of the sulphate of ammonia, when this salt has been used, and also traces of lead, derived from the leaden chambers.

Organic matter is detected by simply dissolving the impure article in water, when a solution of a brownish or even blackish colour is produced, while the pure salt affords a colourless liquid. Sulphates or chlorides are detected by dissolving the salt in water, adding a few drops of nitric acid, and dividing the solution into two portions; to one part chloride of barium is to be added, and to the other a few drops of nitrate of silver solution; a white precipitate in these shows the presence of sulphuric acid in the former, and of hydrochloric acid in the latter.

Hyposulphites are detected by neutralizing a portion of the salt with acetic acid, and adding nitrate of silver; a precipitate is obtained, which is white at first, but soon turns black, in consequence of the oxide of silver being reduced. Lead is detected by transmitting a stream of sulphuretted hydrogen gas through a solution of the salt, which occasions a black precipitate if lead be present.

Commercial carbonate of ammonia is used for cleansing woollen cloths, for the extraction of grease in the manufacture of most fabrics; and when free from organic and mechanical impurities it is used in the preparation of yeast and baking powders.

In agriculture, the salts of ammonia are most valuable, increasing in a remarkable manner the fertility of the land, provided the other constituents peculiar to the crop be present; hence they are to be found in all good manures, the value of which they greatly enhance. Ammonia constitutes one-fifth of the weight of genuine Peruvian guano; stable manure also contains ammonia in large quantities, and it is one of the valuable constituents of bone manure. Ammoniacal salts are extensively used in calico-printing, in the preparation of leather, and in pewtering; for the last process, as well as for tinning sheet-iron and iron vessels, the chloride is preferred; on account of its volatility, it substitutes an ammoniacal atmosphere for the ordinary air, and thus prevents the oxidation of the tin.

ESTIMATION OF AMMONIA.—To the manufacturer who employs ammoniacal salts it is very necessary that he should be able to ascertain their real value, and what amount of work he can perform with their aid. The mode commonly adopted is the determination of the amount of alkali with a standard acid. The salt may likewise be distilled with potash through a LIEBIG's condenser into a flask containing standard acid coloured with a few drops of litmus tincture. The tube from the receiver should not dip into the acid. An U tube containing dilute standard acid up to the bend should be connected with the flask to prevent any loss of ammonia. The liquid is to be kept boiling gently until the drops, as they fall into the acid, cease to render the acid with which they first come in contact blue. The contents of the flask and U tube are then mixed together, and titrated with a standard alkali. The amount of acid which has been neutralized by the ammonia is thus ascertained.

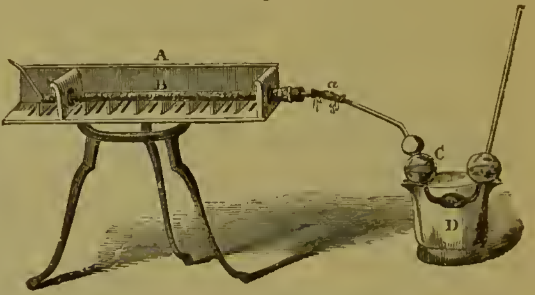
Estimation as Chloride.—In pure solutions of ammonia, or any of its combinations with a feeble volatile acid, the alkali is readily determined by saturating with hydrochloric acid, and evaporating the solution to dryness in a water-bath, till the whole of the free acid and moisture are removed; the dry residue, accurately weighed, gives the amount in the form of ammonium chloride. Should the solution contain only ammonium chloride, evaporation of the water and weighing of the dry residue will give at once the amount of that salt, and the ammonia contained therein may then be calculated. A certain portion of the ammoniacal liquid or solid is weighed, the former in a crucible or covered watch-glass, and the latter in a perfectly clean and dry beaker-glass; these should be rather small, and tared accurately. The solid is then dissolved in water. The succeeding treatment is the same for both. Hydrochloric acid is added in very slight excess, and the solution evaporated carefully, so as to avoid spirting. The evaporation is best conducted in a platinum dish, and when no further loss in weight is sustained by heating in the water-bath, the dish and dry residue are weighed; the difference between this weight and that of the dish is the weight of the chloride of ammonium. If a platinum vessel be not at hand, the evaporation may be continued to dryness in the beaker-glass wherein the solution was originally taken, provided it be small, and the beaker and its contents weighed, as in the foregoing instance; but if this should not happen, when the greater part of the water has been driven off the concentrated liquor is to be carefully transferred to a platinum crucible, or small thin porcelain dish, and also the washings are to be gradually added, the solution evaporated to dryness, and the perfectly dry mass weighed. When the weight of the vessel is deducted from the combined weight, the difference is, as before, the chloride of ammonium. Very accurate results are in this way arrived at, if ordinary care has been exercised, as the trace of chloride which escapes during the evaporation is almost imperceptible. With a similar degree of accuracy, the ammonia may be determined by substituting sulphuric acid for the hydrochloric, and proceeding as above; the ammonia is calculated from the weight of the sulphate.

Estimation as Ammonium Platino-chloride, $(NH_4Cl)_2$, $PtCl_4$. — All those salts of ammonia which are soluble in alcohol may be determined in this form. By converting them into a chloride by the addition of an excess of hydrochloric acid, and evaporating to dryness; then, on dissolving the dry mass in a small quantity of water, and precipitating by a solution of platinic chloride (platinum tetrachloride ($PtCl_4$)) in excess, a yellow crystalline deposit forms, which is the compound salt. The solution is carefully evaporated to dryness at 212° Fahr. (100° C.), the dry mass treated with a little alcohol, and then thrown upon a filter, where it is washed with alcohol till the filtrate ceases to have a yellow colour, which the first washings possess, owing to the excess of bichloride of platinum. Should the filtrate not be coloured, it shows that enough of the precipitant has not been used, and in such a case the operation should be recommenced, taking care to have the platinic chloride in excess, which is known by observing if the supernatant liquid, after the greater part of the crystals has fallen to the bottom, is yellow; the salt is to be thrown upon a filter, and washed with alcohol as above directed. The filter should be carefully deprived of dust, dried at 212° Fahr. (100° C.), till the weight remains constant, before introducing the yellow crystalline compound; and after the substance has been thoroughly dried and weighed, the difference between the latter weighing of the paper and salt, and that of the paper alone, is the ammonium platino-chloride.

As a check upon the determination, the platinum compound may be introduced into a counterpoised porcelain crucible, and heated gently to redness, when all the constituents of the precipitate, except the platinum, are expelled. The metal remains in the form of a black spongy powder, which is weighed, and from its quantity the ammonia contained in the original precipitate ascertained by calculation. During the ignition of the ammonium platino-chloride, the heat must at first be applied very gently, and the crucible kept covered, lest the too rapid evolution of ammonium chloride should carry off some of the platinum mechanically, and give rise to an error. Towards the end of the operation the heat may be increased until no more vapours are observed; the crucible is then allowed to cool, and weighed.

When the ammoniacal compounds are insoluble in alcohol, they are analyzed by a more difficult way than either of the methods already described. The salt is ground with soda lime, and the mixture heated in a combustion tube, in the same way that an organic analysis is conducted. Soda lime is prepared by slaking pure caustic lime with a solution of sodium hydrate of such a strength that one part of the caustic alkali will be equal to two of the lime; it is then heated in a Hessian crucible to dull redness, and, after cooling, pulverized and retained for use in stoppered bottles.

Fig. 19.



Will and Warrentrap's nitrogen bulbs are connected to the end of the tube containing the mixed ammoniacal salts and soda lime by a perforated cork; the bulbs of the apparatus are filled with hydrochloric acid of 1·20 specific gravity, for the purpose of retaining the ammonia which is disengaged from the heated material in the tube. Fig. 19 shows the arrangement as here indicated.

A is the combustion furnace, made of sheet iron (Hofmann's gas combustion furnace is now generally used instead), and resting upon a tripod, or supported upon bricks on a table or cast-iron plate; B, the combustion tube, about 16 inches in length, and made of hard German glass, which bears the high temperature without fusing. The diameter of the tube should be about ⅝ of an inch, and one end should be drawn out to a point, sealed at the extremity, as seen in the engraving. The mouth of the tube is closed by a perforated cork, into which a small tube is introduced, and which is connected to the nitrogen apparatus, C, by means of a caoutchouc connecter, *a*. The nitrogen apparatus has four bulbs, which are filled with hydrochloric acid to the level represented; the apparatus rests upon a convenient support, D. By placing the long limb of the bulbed tube in a small glass filled with the acid, and applying suction to the other end, it is readily filled to the proper height. Great care must be taken that too much acid is not admitted, as then there would be a risk of spoiling the analysis by the liquid flowing back into the combustion tube.

Before proceeding to the determination, the substance should be thoroughly dried, either in the water-bath or over sulphuric acid in the exhausted receiver of an air-pump; 10 to 20 grains are then taken and mixed intimately with the soda lime in an agate or glass mortar, which should be placed upon a large sheet of glazed paper, to prevent the loss of any particles that may be accidentally dropped during the grinding and filling of the tube. As much of the soda lime is put into the tube as is sufficient to fill about ¾ of an inch, and then the mixture of the substance under examination and the lime compound is introduced as speedily as possible. The mortar is rinsed with successive small quantities of the powdered soda lime, and these are added to fill up the empty part of the tube to within 2 inches of its mouth. A loose plug of asbestos is now inserted, and the cork with small tube tightly fitted to it; a space through which the products of the combustion may traverse the tube is made by holding the latter in the hand, and giving it a few gentle taps horizontally against the table; the contents aggregate more closely, and an empty space is formed in the upper part. The tube is now placed in the furnace with the pointed end upwards, as seen in the figure, and the nitrogen apparatus attached by means of the connecter. Care must be exercised that all the joinings are air-tight; this the operator may ascertain by holding a glowing piece of charcoal to the inner bulb, till a few bubbles of expanded air escape to the middle one; if, on removing the coal, the liquid assumes the original level the connections are air-tight. Red-hot pieces of charcoal are now applied to the posterior part of the tube for a short time, to expel the air contained in the apparatus; and after that the pieces are placed at the front end, and gradually added to as the contents become decomposed, till the whole of the tube is at a low red heat. Particular pains should be taken to eliminate the ammonia gradually and steadily, and, above all, not to allow the heat to fall at any part of the tube till all the ammonia contained in that part is expelled; for on removing the coals a vacuum is formed, which causes the reflux of the hydrochloric acid from the nitrogen apparatus into the tube. Hence the coals should be applied to a fresh part before the matter already decomposing is completely exhausted.

When no more ammonia is given off, the pointed end of the tube is broken by a forceps, and suction applied to the long arm of the nitrogen apparatus, in order to draw over the last traces of ammonia that might remain in the apparatus; after which it is detached, and the chloride of ammonium solution transferred to a beaker glass, or platinum dish, together with the water used to rinse the bulbs, and the whole evaporated to dryness.

The residue thus obtained is dissolved in as small a quantity as possible of distilled water, filtered, and the filtrate and washings precipitated by platinic chloride; the solution is then evaporated, and treated with alcohol. The remainder of the operation is conducted in the same way as already described. The results, when the operation is carefully performed, are very accurate.

The soda-lime process may be shortened by taking a known weight of pure hydrochloric acid, the quantity of real acid in which may be ascertained either by specific gravity or by an acidimetrical examination. In this case, after the ammonia from the mixture in the combustion tube has been absorbed, the contents of the nitrogen apparatus are transferred to a beaker glass together with the washings, and the excess of acid neutralized with a standard solution of ammonia.

PELIGOT determines this excess of acid by dissolving caustic lime in a solution of sugar, and neutralizing the acid with the solution thus obtained. The liquid is preserved from contact with carbonic acid in well-stoppered bottles.

The saccharate of lime thus formed has an alkaline reaction, and neutralizes acids like an alkaline carbonate. Its saturating power is tested with a dilute solution of pure concentrated sulphuric acid.

Estimation by Bromized Sodium Chloride.—A standard solution of strongly alkaline sodium chloride containing bromine sets free nitrogen when poured into a solution of ammonia. To prepare the solution:—Dissolve 1 part of sodium carbonate in 15 parts of water, cool the liquid with ice, and saturate it with chlorine whilst still cool, and add sufficient strong solution of sodium hydrate to render the whole caustic. Before using, add bromine in the proportion of 2 to 3 grains to the litre. (50 c.c. of this solution will decompose fully 2 grains of ammonium chloride). The solution is titrated with a solution of sodium arsenite containing 4·95 grm. As_2O_3 in 1 litre (1 c.c. = ·000566 grm. NH_3) (see CHLORINE).

Estimation by Nessler's Solution (see WATER).—When NESSLER's solution (a solution of mercuric iodide in potassium iodide and potassium hydrate) is added to a certain volume of fluid containing a very minute unknown quantity of ammonia, a certain tint is produced. And the unknown quantity of ammonia may be determined by imitating this tint with a known quantity of ammonia.

The solutions used are:—1. A standard solution of ammonia (1 c.c. = 1 mgrm. NH_3). This is prepared by dissolving ·315 grm. of ammonium chloride in 1 litre of water. 2. NESSLER's solution.

Potassium iodide,	3·5 grm.
Mercuric chloride,	1·6 grm.
Water,	40 c.c.
Potassium hydrate solution,	*quant. suff.*

The potassium iodide is dissolved in 10 c.c. of water, the mercuric chloride in 30 c.c. of water. The latter solution is then gradually added to the former till a permanent precipitate is produced. Sufficient potash solution is then added to make the fluid measure 100 c.c.

The test glasses should be colourless and of equal size. They should be marked at 100 c.c., and the mark should be at the same height in each cylinder.

The process is carried out thus :—The solution to be tested is placed in one of the cylinders, made up to 100 c.c. with water, and 1·5 c.c. of Nessler's solution mixed with it by means of a pipette. The tint produced is carefully observed, and then imitated as nearly as possible by running into the other cylinder as many tenths of a c.c. of ammonia as it is imagined will produce the same effect. The second cylinder is then filled up to 100 c.c. with water, 1·5 c.c. of Nessler's solution added, and after allowing the whole to stand a few minutes, the tints of the contents of the two cylinders compared. If not alike, a fresh experiment is necessary. When the solution contains more than 1 mgrm. of ammonia in 100 c.c., the tint produced is far too dark for correct estimation.

ANÆSTHETICS (French, *anesthésiques*; from ἀ priv., and *αἰσθάνομαι*, to feel) is the name given to certain substances which, when introduced into the circulation, are capable of depriving the patient of the power of motion and of sensation. These agents are now largely used in therapeutics and in dentistry, for producing insensibility to the pain necessarily attendant upon surgical operations, and also as remedial agents in certain diseases characterized by great excitement of the sensorial functions.

Although it had long been known that narcotics, when administered in sufficiently large doses, produced coma and insensibility, the use of anæsthetics for the alleviation of pain is of comparatively recent date. An American, Dr. Jackson, first suggested their use in 1846, and immediately afterwards they were successfully employed in dentistry by Dr. Morton, and also by the American surgeons, Warren, Biglow, and Hayward. On the 19th of December in the same year Liston, the eminent surgeon, and Mr. Robinson, a dentist, operated on patients rendered insensible by the inhalation of ether vapour. In 1847 ether was employed in England and in France, and towards the end of that year Flourens pointed out the anæsthetic properties of chloroform; to Dr. Simpson of Edinburgh, however, we are indebted for the introduction of the use of chloroform in surgical and obstetrical practice. Dr. Snow in 1849 published a work on the inhalation of ether, and also discovered that amylene was capable of producing effects similar to those of chloroform; the latter, however, is now almost invariably used in surgical operations, as its action is much more rapid than that of ether. Amylene is intermediate in rapidity of action between chloroform and ether; but as two out of the few cases in which it was tried proved fatal, its use was discontinued. Methylene dichloride, also, has recently been recommended by Dr. Richardson as an anæsthetic, but it has the disadvantage of causing great depression.

These agents are administered through the respiratory organs by inhaling the vapour mixed with air. For this purpose some of the chloroform is poured on to a sponge or handkerchief, which is then applied to the mouth and nostrils of the patient in such a manner that the air which passes into his lungs is saturated with the vapour. Ether is administered in the same way. Dr. Snow, who has paid much attention to this matter, invented a special form of apparatus for inhaling the vapour.

The first effect produced by the administration of the anæsthetic agent is a species of intoxication caused by its action on the cerebral lobes, and as this action extends to the cerebellum the patient becomes incapable of directing his movements—a result similar to that of alcoholic intoxication. In the next stage the spinal cord is attacked, unconsciousness supervenes, and all powers of motion and sensation are lost. The individual is now said to be in a state of *anæsthesia*; but the heart beats, breathing continues, and the other essential functions of the body are carried on. If, however, the exhibition of the anæsthetic agent is still continued, the temperature of the body becomes lowered, the movements of respiration and circulation become impaired, the heart ceases to beat, and death finally ensues.

The introduction of anæsthetics into surgical practice has greatly contributed to its progress: the patient being motionless and free from pain, the operator is enabled to perform with much greater delicacy and readiness; moreover, in the reduction of dislocations and of hernia, the muscles being flaccid, the obstacle produced by their contraction is removed.

M. Velpeau endeavoured to produce *local anæsthesia*, or insensibility of that part of the body to be operated on, by means of a refrigerating mixture of ice and salt; it was, however, found to be very unmanageable. Since then local anæsthesia has been successfully obtained by means of a spray of ether directed upon the part; the intense cold produced by the rapid evaporation of the ether entirely depriving it of sensation.

See Chloroform, Ether, and Nitrous Oxide or laughing gas, for methods of preparation and properties. Various other agents, such as amylene, ethyl chloride, ethyl nitrate, benzene, and carbon bisulphide, have been proposed as anæsthetics; but from their injurious effects they have never been brought into use.

ANILINE AND ANILINE DYES—Aniline. The term Aniline, or Aniline oil, is used commercially to indicate a variable mixture of bodies, amongst which certain members of the series of organic bases known as the *Aniline homologues* greatly preponderate. Aniline itself, the parent member of this series, is indicated by the formula $C_6H_5.NH_2$; the formulæ of all the true homologues of aniline are deducible from this parent symbol by substituting for one or more of the five H symbols in the radical C_6H_5 a corresponding number of univalent hydrocarbon radicals of the methyl series, just as the formulæ of benzene homologues are deducible from the symbol of benzene by a similar process. (*Vide* article, BENZOL.) The main constituents of commercial aniline oils consist of the lower members of this series, viz. :—

Name of Base.	Rational Formula.	Dissected Formula	Boiling Point. Degs. C.	Melting Point.
Aniline, ...	C_6H_7N	$C_6H_5\ \ NH_2$	182°	—8°
Para-toluidine, ..	C_7H_9N	$C_6H_4\left\{\begin{matrix}NH_2\\(CH_3)_p\end{matrix}\right.$	200°	+ 45°
Meta-toluidine, ..	C_7H_9N	$C_6H_4\left\{\begin{matrix}NH_2\\(CH_3)_m\end{matrix}\right.$	199°	{liquid at —20°
Various Xylidines, ...	$C_8H_{11}N$	$C_6H_3\left\{\begin{matrix}NH_2\\CH_3\\CH_3\end{matrix}\right.$	{212° to 216°	—
Various Cumidines, ... &c., &c.	$C_9H_{13}N$	$C_6H_2\left\{\begin{matrix}NH_2\\CH_3\\CH_3\\CH_3\end{matrix}\right.$	About 225°	Some liquid some solid, one melts at 60°.

Just as various isomeric modifications of certain homologues of benzene are known, the formulæ of which are distinguished one from the other by special devices in their "dissection:" so various isomeric modifications of the higher aniline homologues are known, denoted by analogous methods. Comparatively few of these have as yet been submitted to special study; but in the toluidine series all three of the isomerides predictable in accordance with the symbolic system of KEKULÉ for the notation of benzene derivatives are actually known (*vide* article BENZOL), although only two (para- and meta- toluidine), have as yet been shown to exist in commercial aniline oils.

In addition to these bodies, the aniline oils of commerce usually contain small quantities of other substances, for the most part formed as bye-products during the manufacture of the oils; the chief of these constituents are :—Diamidobenzene, $C_6H_4(NH_2)_2$, Diamidotoluene, $C_6H_3\left\{\begin{matrix}CH_3\\(NH_2)_2\end{matrix}\right.$; higher homologues thereof:—Paraniline, $C_{12}H_{14}N_2$; Azobenzene, $C_{12}H_{10}N_2$; higher homologues thereof:—Xenylamine, $C_6H_4\left\{\begin{matrix}C_6H_5\\NH_2\end{matrix}\right.$; Acetanilide, $\left.\begin{matrix}C_6H_5\\C_2H_3O\\H\end{matrix}\right\}N$; Para acet-toluide, $\left.\begin{matrix}C_6H_4(CH_3)_p\\C_2H_{30}\\H\end{matrix}\right\}N$; Meta acet-toluide, $\left.\begin{matrix}C_6H_4(CH_3)_m\\C_2H_3O\\H\end{matrix}\right\}N$, &c.

ANILINE (*par excellence*), AMIDOBENZENE, or PHENYLAMINE.—This base was first discovered by UNVERDORBEN in 1826, as a product of the dry distillation of indigo; on account of the facility with which its salts crystallize, he applied the term *Crystalline* to it. In 1834, RUNGE obtained from coal tar a substance to which he gave the name *Kyanol*, on account of its power of producing purple or violet-blue tints with bleaching powder. In 1840, FRITZSCHE found that a volatile liquid base is produced, by acting on indigo and certain of its derivatives, with caustic potash; to this he gave the name ANILINE, from *anil*, the Portuguese for indigo. Soon after ZININ obtained a substance, which he termed *Benzidam*, by reducing nitrobenzene by alcoholic ammonium sulphide, and FRITZSCHE showed that this substance was identical with his aniline. In 1843, HOFMANN showed that crystalline, kyanol, aniline, and benzidam are one and the same substance. In 1856, PERKIN discovered the production of mauve by the oxidation of aniline sulphate by potassium dichromate; this process he subsequently patented. The manufacture of aniline speedily became a recognized industrial operation, the cheapest source being nitrobenzene, which was derived from the benzene occurring in coal tar. The distillation of the latter hydrocarbon from the waste products of gas works is now a distinct trade (*vide* article COAL TAR DISTILLATION). The discovery of fuchsine by VERGUIN, in 1858, as a product of the action of certain dehydrogenizing agents on commercial aniline oils, greatly stimulated the growth of this industry; and the subsequent discoveries of triphenyl rosaniline (Bleu de Lyons), ethylated and methylated rosanilines (HOFMANN'S blues and violets), aldehyde green, iodine green, violets de Paris, diphenylamine blue, &c., &c., added rapidly in succession to the magnitude and importance of the trade in colours derived from aniline oils as starting point. The value of the aniline dyes now yearly manufactured, is estimated by VERSMANN to exceed £2,000,000 sterling.

The chief processes by which aniline can be formed are these :—

A. From BENZENE.

1. By the passage of a mixture of benzene and ammonia through red hot tubes—(Berthelot).

$$\underset{\text{Benzene.}}{C_6H_6} + \underset{\text{Ammonia.}}{NH_3} = H_2 + \underset{\text{Aniline.}}{C_6H_5,NH_2}.$$

2. By the conversion of benzene into nitrobenzene and subsequent reduction by nascent hydrogen, or by other reducing agents equivalent thereto.

$$C_6H_6 + NO_2,OH = H,OH + C_6H_5,NO_2$$
$$C_6H_5,NO_2 + 3H_2 = 2H_2O + C_6H_5,NH_2.$$

The following are the principal methods by which the reduction may be effected :—

a Action of alcoholic sulphuretted hydrogen or sulphides—(*Zinin*).
b " acetic acid and metallic iron—(*Béchamp*).
c " zinc and alcoholic hydrochloric acid—(*Hofmann*).
d " zinc dust and hot water—(*Kremer*).
e " tin and hydrochloric acid—(*Scheurer Kestner*).
f " hydriodic acid, at 104° C.—(*Mills*).
g " caustic soda and grape sugar—(*Vohl*).
h " alkaline arsenites—(*Wöhler*).
i " stannous chloride—(*Kekulé*).
k Distillation with alcoholic potash, *inter alia*—(*Mitscherlich*).
l Passage through the animal organism—(*Letheby*).
m Action of iron filings and very dilute hydrochloric acid—(*Brimmeyr*).
n Water and particles of iron covered superficially with copper, by means of a short immersion in copper sulphate solution—(*Coblentz*).
o Water and solution of cuprous oxide in ammonia—(*Wagner*).
p Water and carbon disulphide at 160° C.—(*Schlagdenhauffen*).

B. From PHENOL.

1. By heating with ammonia at 300° C., for two or three weeks—(LAURENT, HOFMANN).

$$C_6H_5,OH + NH_3 = H_2O + C_6H_5,NH_2.$$

(Berthelot finds that only traces of aniline are thus formed after heating to 280°C. for twenty-four hours, a little more being produced at 360° C.)

2. By transformation into phenyl chloride and heating with ammonium hydrochloride—(DUSART and BARDY). GIRARD and DE LAIRE have been unable to verify this reaction.

C. From AMIDOBENZOIC ACIDS AND CERTAIN OF THEIR ISOMERIDES BY HEATING, EITHER ALONE OR TOGETHER WITH AN ALKALI.

1. Amidobenzoic acid (Anthranilic acid)—

$$C_6H_4\left\{\begin{matrix}CO,OH\\NH_2\end{matrix}\right. = CO_2 + C_6H_5,NH_2$$

2. Salicylamide—

$$C_6H_4\left\{\begin{matrix}OH\\CO,NH_2\end{matrix}\right. = CO_2 + C_6H_5,NH_2$$

—(*Hofmann and Muspratt*).

3. Nitrotoluene—

$$C_6H_4\left\{\begin{matrix}NO_2\\CH_3\end{matrix}\right. = CO_2 + C_6H_5NH_2$$

—(*Hofmann and Muspratt*).

D. From OTHER SOURCES.

Destructive distillation of indigo—(*Unverdorben*).
" " coal—(*Runge*).
" " turf—(*Vohl*).
Action of caustic potash on indigo, isatin, &c.—(*Fritzsche*).

Of these reactions only those falling under class A can be made commercially available, the extraction of the aniline ready formed in coal and other tars being too costly. Out of these processes only 2. *a*, *b*, *c*, *d*, *g*, and *h*, appear to have been used on anything like a manufacturing scale; process 2. *b* (BÉCHAMP'S) being the one now almost universally employed, not only for the preparation on the large scale of aniline itself, but also of the mixtures known as aniline oils.

Pure aniline is a perfectly colourless oily fluid, which refracts light strongly, and has a weak aromatic odour; it is but sparingly soluble in water, but is dissolved with ease by ether, alcohol, carbon disulphide, and hydrocarbons. It boils at 182° C., and solidifies in a freezing mixture to a crystalline mass melting at —8° (LUCIUS: this only takes place when the substance is exceedingly pure, ordinarily pure aniline not solidifying at —20° C.): its specific gravity at 16°C. is 1·02. By exposure to air and light it becomes brown, probably by oxidation. Its basic properties are well-marked, a lengthy series of well-defined salts being obtainable from it; it has scarcely any action on vegetable colours, not affecting either turmeric or litmus, although it turns violet dahlia tincture green: when boiled with solution of ammoniacal salts it expels ammonia, forming aniline salts; cold solution of ammonia, however, precipitates aniline from its salts. Aniline throws down the hydrates from solutions of many metallic salts, *e.g.*, iron, zinc, alumina, &c., but not of others, *e.g.*, manganese, cobalt, nickel, lead, and chromium. It is a powerful narcotic poison.

Commercially pure aniline is prepared as in the manufacture of aniline oils generally; it may also be obtained from the lighter kinds of commercial oils by successive distillations, the first runnings being in each case collected apart and redistilled. For the preparation of certain dyes an oil containing ninety-five per cent. at least of pure aniline, and not more than five per cent. of the higher homologues, is required. The separation of the higher homologues is considerably facilitated by the addition of from ten to twenty per cent. of sulphuric acid to the mixture of bases before distilling, collecting apart the distillate at 182° to 185° C., and treating this repeatedly in the same way.

TOLUIDINE.—Three isomeric modifications of this base are known, distinguished as para-, meta-, and ortho-toluidine respectively.

Paratoluidine.—$C_7H_9N = C_6H_4\left\{\begin{matrix}NH_2\\CH_3.\end{matrix}\right.$ This was discovered by HOFMANN and MUSPRATT in 1845, being derived from the action of reducing agents on nitrotoluene, obtained by nitrating toluene obtained from balsam of tolu. CHAUTARD also obtained it in small quantity by acting with caustic potash on the yellow resin formed when turpentine is oxidized by nitric acid. It is also formed by the reducing action of hydriodic acid at 180°-200° C. on pure nitrobenzoic acid, or on the amidobenzoic acid, derived thence by reduction (ROSENSTIEHL)—

$$C_6H_4\left\{\begin{matrix}NH_2\\CO,OH\end{matrix}\right. + 6HI = 3I_2 + 2H_2O + C_6H_4\left\{\begin{matrix}NH_2\\CH_3\end{matrix}\right.$$

and by heating methylaniline hydrochloride in sealed tubes to 350° for a day (HOFMANN)—

$$C_6H_5,NH,CH_3 = C_6H_4\left\{\begin{matrix}NH_2\\CH_3.\end{matrix}\right.$$

This reaction is one of the class where "change of position," in the various groups of symbols that make up the dissected formulæ of the materials and products is said to occur.

When perfectly pure, paratoluidine melts at 45° C., and boils at a temperature variously stated at from 198° to 200° C., by different observers. Minute quantities of foreign substances greatly lower its melting point; hence 40° C. was a long time considered as the true melting point. In chemical character it closely resembles aniline.

Pure paratoluidine is not commercially manufactured; it may be obtained by reducing paranitrotoluene (*vide* article BENZOL) by nascent hydrogen, and crystallizing the resulting product from alcohol; or if larger quantities are required, it may be extracted from those aniline oils that boil near 200° C. by the following process, described by BRIMMEYR, viz.: fractional distillation, the portions boiling between 195° and 205° C. being collected apart; this fraction is then heated with half its weight of oxalic acid and four times its weight of water, until solution is effected; the hot liquor is then cooled to 80° C. and rapidly filtered: crystals of paratoluidine oxalate separate, from which the base is obtained by pressing, and then boiling with ammonia solution to which enough alcohol has been added to yield a clear solution of paratoluidine. On cooling crystals of the base form, which may be recrystallized from alcohol (or better, petroleum distillates boiling at 80°–100° C. —H. Müller); the oxalate may be recrystallized with advantage before the separation of the free base (Noad).

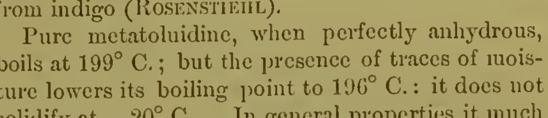
Metatoluidine.—Rosenstiehl discovered in 1868 that the substance boiling at 198° C., and possessing the composition C_7H_9N, occurring in ordinary aniline oils, consisted mainly of a mixture of paratoluidine and an isomeride, since recognized as metatoluidine, but termed *pseudotoluidine* by its discoverer. This mixture exhibits the composition and boiling point of paratoluidine, but refuses to crystallize at ordinary temperatures; at 0° C., however, and in presence of a few drops of water, or a crystal of paratoluidine, much solid paratoluidine separates; by filtration and pressure this is separated, and the yet liquid mixture of bases is again treated in the same way. The portion finally remaining liquid is converted into neutral oxalate and then digested with anhydrous ether, in which menstruum paratoluidine oxalate is almost insoluble, whilst metatoluidine oxalate dissolves readily therein (aniline oxalate is but slightly soluble in anhydrous ether). The ethereal solution is evaporated, and the residue again treated with ether; finally, the metatoluidine oxalate thus separated is crystallized from water or alcohol, and decomposed by caustic soda.

The relations between metatoluidine and other benzene derivatives have been studied by Rosenstiehl, Beilstein and Kuhlberg, Huebner and Wallach, Körner, Wroblewsky, and others, from whose researches it results that when toluene is nitrated two isomeric nitrotoluenes are formed by the reaction. One of these is solid, and forms paratoluidine only on reduction; the other is liquid, and produces metatoluidine. On the other hand, either para- or meta- toluidine, when treated with nascent hydrogen evolved by heating with strong hydriodic acid, gives rise to *absolutely the same toluene* in each case, this toluene being in either instance capable of producing both solid and liquid nitrotoluene on nitration. Hence it results that commercial aniline oil necessarily contains both para- and meta- toluidine, produced by the reduction of the para- and meta- nitrotoluenes simultaneously formed by the nitration of the toluene contained in commercial benzol: only one toluene, however, exists in this substance (*vide*, article Benzol).

Metatoluidine is also obtained by the following processes:—Reduction of meta-nitrotoluene resulting from the decomposition of the diazo-derivative of the nitrotoluidine formed by the reduction of dinitrotoluene melting at 70·5, produced by the continued action of nitric acid on toluene (Beilstein and Kuhlberg). Passage of the vapour of the amidotoluic acid, resulting from the action of nitric acid on xylene, over soda lime (Beilstein and Kuhlberg):—

$$C_6H_3\left\{\begin{array}{l}NH_2\\CO,OH\\CH_3\end{array}\right. = CO_2 + C_6H_4\left\{\begin{array}{l}NH_2\\CH_3\end{array}\right.$$

Action of nascent hydrogen (zinc and hydrochloric acid, sodium amalgam, &c.) on the nitrobromotoluene resulting from the action of strong well-cooled nitric acid on solid bromotoluene melting at 25°·4 C. (Körner, Rosenstiehl and Nikiforoff.)



$$C_6H_3\left\{\begin{array}{l}NO_2\\Br\\CH_3\end{array}\right. + 4H_2 = HBr + 2H_2O + C_6H_4\left\{\begin{array}{l}NH_2\\CH_3\end{array}\right.$$

Metatoluidine also occurs in the aniline obtained from indigo (Rosenstiehl).

Pure metatoluidine, when perfectly anhydrous, boils at 199° C.; but the presence of traces of moisture lowers its boiling point to 196° C.: it does not solidify at —20° C. In general properties it much resembles paratoluidine, but differs in its crystalline form and in the solubility of its salts; it also gives rise to derivatives which exhibit considerable difference in boiling and melting points, &c., from the corresponding derivatives of paratoluidine. A marked difference in the action of bleaching powder on the two serves as a useful qualitative test; if the solution oxidized by bleaching powder be shaken up with ether, and the ethereal extract agitated with acidulated water, no result ensues in the case of paratoluidine (nor of aniline), but a magnificent tint resembling that of permanganate solution is developed with metatoluidine.

Rosenstiehl employs three methods for the separation of metatoluidine from commercial toluidine, viz.:—A process depending on the different solubility of the acid oxalate, the paratoluidine salt requiring 125 parts of water for solution, whilst the metatoluidine salt is much more soluble; one taking advantage of the tendency possessed by the hydrochloride of each base to yield supersaturated solutions, from a mixture of which the one salt or the other can be made to crystallize by dropping in a crystal of the required kind; and a method consisting in fractional saturation with an acid, preferably sulphuric acid—paratoluidine is first taken up, and metatoluidine can then be distilled off by means of steam.

Bindschleder gives the following commercial process founded on Rosenstiehl's observations:—Commercial "toluidine" (a mixture of para- and meta- toluidine with a small percentage of aniline) is distilled, the portion passing at 198° C. being collected apart; to 200 kilos. of this, 25 litres of water are added, in which 2½ kilos. of oxalic acid have been previously dissolved; 6 litres of hydrochloric acid at 20° Beaumé are then added, and the whole boiled and cooled to 60° C. and rapidly filtered; paratoluidine oxalate is thus thrown down. To the filtrate 2 kilos. of oxalic acid are then added, whereby a mixture of oxalates of para- and meta-toluidine is precipitated, from which the bases are regained by means of soda, and the mixture is then used over again in the first part of the process. The filtrate is distilled with soda, when metatoluidine is obtained sufficiently pure for manufacturing purposes. (Berichte der Deut. Chem. Ges., vol. vi. p. 448.)

Schad separates metatoluidine from aniline oils boiling near 200° C., by adding to every 10 lbs. of oil, nitric acid of specific gravity 1·2, in sufficient quantity to convert it into nitrate (quantity not stated). The hot liquid is cooled and well stirred, and the crystalline mass thoroughly expressed; the solid residue is dissolved in as much boiling water as will give a solution of sp. gr. 1·1 (while hot?), the solution cooled and stirred. The crystals formed are again pressed out and dissolved in boiling water to sp. gr. 1·075, allowed to crystallize, and again expressed, and finally again dissolved to sp. gr. 1·05, crystallized and expressed. These last crystals are nearly pure metatoluidine nitrate, from which the free base is readily obtained by distillation with soda. Higher homologues are still present to some extent, but these are separated by converting the mixture into hydrochlorides, heating with water as before. Finally, a metatoluidine boiling constantly at 197° C., is obtained, the yield being about 1 lb. or 10 per cent. of the aniline oil used.

Commercial toluidine prepared by COUPIER, and boiling constantly at 198° C., was found by ROSENSTIEHL to contain:—Paratoluidine, 62 p.c.; metatoluidine, 36 p.c.; aniline, 2 p.c. On the other hand, commercially pure aniline contained:—Aniline, 95 p.c.; mixture of para- and meta- toluidine, 5 p.c. It does not appear that the ratio between the para- and meta- toluidine present in commercial aniline oils is at all constant; slight differences in the circumstances attending the nitration of the hydrocarbons employed naturally produce differences in the relative quantities of para- and meta- nitrotoluene formed *inter alia*.

Orthotoluidine.—The existence of this third isomeride in commercial aniline oil has not yet been definitely proved, although it is not improbable. It was obtained by BEILSTEIN and KUHLBERG by the reduction of the orthonitrotoluene formed by decomposing with alcoholic potash the nitro-acet-toluide produced by nitrating para-acet-toluide, and converting the resulting nitrotoluidine into nitrotoluene by GRIESS' process (*vide* article BENZOL).

The following table illustrates the differences between these three isomeric toluidines:—

	Paratoluidine.	Metatoluidine.	Orthotoluidine.
At ordinary temperature, ...	Solid.	Liquid.	Liquid.
Melting point,...	+ 45	Does not solidify at—20°	Does not solidify at—13°
Boiling point,....	198°, various observers. 200°, Beilstein and Kuhlberg.	198° Rosenstiehl, 199° Beilstein and Kuhlberg.	197° Beilstein and Kuhlberg.
Specific gravity,	Nearly identical with water. 0·9965 Girard and De Laire. 1·0017, other observers.	1·0002 at 16·3, Rosenstiehl.	0·998 at 25°, Beilstein and Kuhlberg.
Colour reaction with bleaching powder & ether, &c.,.................	Nil.	Permanganate tint.	Permanganate tint.
Melting point of acet-toluide,.....	147°	102°	65° to 65·5°
Boiling point of acet-toluide,.....	300° to 307°	295°	302° to 304°
Solubility of acet-toluide,.....	1000 water dissolve 0·886 at 22°	1000 water dissolve 8·5 at 19°	1000 water dissolve 1·3 at 13°

Xylidine.—The aniline homologues from the reduction of the nitroxylenes in commercial nitrobenzol have not been minutely studied. HOFMANN and MARTIUS extracted from aniline tailings, *queues d'aniline*, a xylidine boiling at 212° C.; and TAWILDAROW prepared from nitro-isoxylene, a xylidene boiling at 216° C., sp. gr. 0·935 at 18°·5 C. The xylidine present in aniline oils is probably a mixture of the product of TAWILDAROW and the corresponding body derived from nitro-paraxylene. CAHOURS gives 214° C. as the boiling point of coal tar xylidine, and BEILSTEIN 215° C.

Besides these several other isomerides are known, but are probably not contained in aniline oils.

Cumidine.—SCHAFER has obtained from the nitrocumene derived from pseudo-cumene (trimethyl benzene) which melts at 71° C., a cumidine which crystallizes in needles melting at 60° C., whilst FITTIG and STORER have obtained from the nitrocumene derived from mesitylene, a cumidine which does not solidify at 0° C.; to this base the term *mesidine* is applied. Probably the cumidine occurring in aniline consists of a mixture of both of those isomerides and possibly others; the cumidine obtained from coal tar cumene (mixture of isomerides) is liquid at ordinary temperatures, and boils at 225° C.

Besides the aniline series of bases, commercial aniline oils also contain smaller or larger quantities of the following and other substances:—

Diamidobenzene (*phenylene-diamine*).—By the reduction of dinitrobenzene occurring in the nitrobenzol used, there is produced, firstly, a nitraniline, and secondly, a diamidobenzene.

$$C_6H_4\begin{cases}NO_2\\NO_2\end{cases} + 3H_2 = 2H_2O + C_6H_4\begin{cases}NH_2\\NO_2\end{cases}$$

$$C_6H_4\begin{cases}NH_2\\NO_2\end{cases} + 3H_2 = 2H_2O + C_6H_4\begin{cases}NH_2\\NH_2\end{cases}.$$

Three diamidobenzenes are actually known, only one of which occurs in aniline oils, viz., that derived from the reduction of the dinitrobenzene formed by the continued action of nitric acid on benzene. It is a solid crystalline body melting at 63° C. and boiling at 287°C.; by the action on this of nitrous acid, phenylene brown, $C_{12}H_7N_2(NH_2)_3$, is formed. (*vide* Aniline brown).

Diamido-toluene.—By the further nitration of paranitro-toluene there is formed a dinitrotoluene melting at 71° C. (*vide* BENZOL). By the action of reducing agents a nitrotoluidine and a diamidotoluene are formed:—

$$C_6H_3\begin{cases}CH_3\\NO_2\\NO_2\end{cases} + 3H_2 = 2H_2O + C_6H_3\begin{cases}CH_3\\NH_2\\NO_2\end{cases}$$

$$C_6H_3\begin{cases}CH_3\\NH_2\\NO_2\end{cases} + 3H_2 = 2H_2O + C_6H_3\begin{cases}CH_3\\NH_2\\NH_2\end{cases}.$$

This diamido-toluene melts at 99° C. and boils at 300° C. Diamido-xylenes and diamidocumenes are also probably present in high boiling aniline oils.

Paraniline.—HOFMANN has shown that aniline tailings (*queues d'aniline*) contain a solid body melting at 192° C. and boiling above 330° C. Beyond the fact that this substance is indicated by a formula double of that applied to aniline, little is known of its relation to that body or of its mode of formation.

Xenylamine.—$C_{12}H_9,NH_2$. HOFMANN has also found in aniline tailings a base which has the composition and properties of amidodiphenyl, *i.e.*, is related to diphenyl, C_6H_5,C_6H_5, as aniline is to benzene; it is a crystalline body melting at 45° and boiling at 322° C.

Azobenzene or *Azobenzide.*—This body, $C_{12}H_{10}O_2$, is formed from nitrobenzene by the action of a quantity of hydrogen insufficient to transform it into aniline:—

$$2C_6H_5,NO_2 + 4H_2 = 4H_2O + C_{12}H_{10}N_2.$$

By the action of nascent hydrogen it yields aniline, *inter alia*; conversely, certain oxidizing agents transform aniline into azobenzene. Nascent hydrogen also forms a body related to xenylamine just as diamidobenzene is to aniline, viz., *Benzidine* or *xenylene diamene* (diamidodiphenyl)—

$$C_{12}H_{10}N_2 + H_2 = C_{12}H_8\left\{\begin{matrix}NH_2\\NH_2\end{matrix}\right.$$

This latter substance is probably therefore a constituent of aniline oils. Azobenzene melts at 65°, and distils unchanged at 293° C., whilst benzidene melts at 118° C., and is partially decomposed by distillation.

Azotoluene.—This body is related to toluene, just as azobenzene to toluene; probably more than one modification exists in aniline oils. Bodies similarly related to the nitroxylenes and nitrocumenes are also likely to be present in the higher boiling aniline oils.

Acetanilide.—This body is formed by the action of heat on aniline acetate and in several other ways—

$$C_6H_5,NH_2,C_2H_3O,OH = H_2O + C_6H_5,NH,C_2H_3O$$

It forms a crystalline mass melting at 101° C. (GREVILLE WILLIAMS); 106°·5 C. (STAEDELER); 111° C. (GERHARDT); 112°–113° C. (MERZ and WEITH); and boiling at 295° C. Dilute alkaline solutions have little action on it, for which reason, if once formed in BÉCHAMP'S process, it is apt to remain unaltered, and to lead to a considerable loss of aniline; fusion with caustic potash, however, readily transforms it into aniline and potassium acetate:—

$$C_6H_5,NH,C_2H_3O + KOH = C_6H_5,NH_2 + C_2H_3O,OK$$

Acet-toluide.—Two isomerides of this character occur in aniline oils, viz., the para- and meta-toluidine derivatives; these melt at 147° and 102° C., and boil at 306° and 295° C. respectively. Higher homologues, *e.g.*, acetoxylides, acetocumides, &c., are known, and without doubt are present in the higher boiling aniline oils.

It is wholly unknown whether any of the foregoing constituents of aniline oils, other than aniline and its homologues, have any influence on the tinctorial properties of the aniline colours made from oils containing them; for the most part these bodies are removed by fractional distillation of the aniline oil before it is used for fuchsine, and especially for methyl violet; still the presence of even traces may not improbably somewhat modify the nature of the reactions taking place, and may consequently have some influence on the character of the dye-stuff produced, and on its yield. It happens in several instances that whilst the action of certain reagents on a single homogeneous aniline base gives rise to no product possessed of tinctorial properties, the same substance produces valuable dyes when made to act on a mixture of bases nonproductive when separately treated. Thus the oxidizing materials which produce rosaniline when a mixture of aniline and paratoluidine is acted on, give rise to no red colour with pure aniline (HOFMANN), to a violet colouring matter, *violaniline* (GIRARD, DE LAIRE, and CHAPOTEAU). A red very similar to rosaniline, but differing from it in certain respects, is produced when aniline and metatoluidine are similarly treated; whilst a mixture of para- and meta-toluidine gives a third red under the same circumstances. On the other hand, neither para- nor meta-toluidine gives any red colour when subjected alone to this treatment (ROSENSTIEHL, HOFMANN). Similarly, pure xylidine gives no red, whilst a mixture of xylidine and aniline gives a fine crimson red dye (HOFMANN). Again, azobenzene has been shown by STAEDELER to form blue and violet colouring matters when heated with aniline or toluidine hydrochloride, the latter also yielding a ruby red compound. It may therefore readily happen, that the presence of a minute quantity of a body, inert as a dye-producer itself, may greatly affect the quality of the dye formed from the other substances simultaneously present; and it is by no means improbable that the success of various devices and "secret processes" practised by colour makers depends on such circumstances, the exact nature of the influence being unknown, and the process being arrived at in an empirical way.

Substituted Anilines.—Of the numerous bodies known from the researches of HOFMANN to be obtainable from aniline and its homologues by substitutive reactions, only that class is of commercial interest where the substitution is of a kind that gives rise to a secondary or a tertiary monamine, *i.e.*, which is symbolically indicated by the replacement of one or both of the H symbols in the NH_2, radical by the symbols of some hydrocarbonous radical: thus,

$$C_6H_5,NH_2 + CH_3I = HI,C_6H_5,NH,CH_3$$

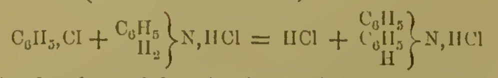
$$C_6H_5,NH,CH_3 + C_2H_5Br = HBr,C_6H_5,N\left\{\begin{matrix}CH_3\\C_2H_5\end{matrix}\right.$$

Of the bodies thus producible, the following are the most important:—

Diphenylamine, $\left.\begin{matrix}C_6H_5\\C_6H_5\\H\end{matrix}\right\}N$. This base is obtainable by the dry distillation of certain aniline dyes, *e.g.*, rosaniline, leucaniline, &c. (HOFMANN), and by the action of aniline on aniline salts (*e.g.*, the hydrochloride), or on a mixture of materials which give rise to the formation of an aniline salt, *e.g.*, aniline and a phenyl sulphonate (GIRARD, DE LAIRE, and CHAPOTEAU), the corresponding ammonium salt being formed

$$\left.\begin{matrix}C_6H_5\\H\\H\end{matrix}\right\}N + \left.\begin{matrix}C_6H_5\\H\\H\end{matrix}\right\}N,HCl = \left.\begin{matrix}C_6H_5\\C_6H_5\\H\end{matrix}\right\}N + \left.\begin{matrix}H\\H\\H\end{matrix}\right\}N,HCl$$

Also by that of nascent phenyl chloride on aniline hydrochloride (prepared by heating together phenol, sal-ammoniac, and hydrochloric acid), or by passing phenyl chloride or iodide and aniline vapour through a hot tube (DUSART and BARDY).



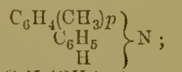
$$C_6H_5,Cl + \left.\begin{matrix}C_6H_5\\H_2\end{matrix}\right\}N,HCl = HCl + \left.\begin{matrix}C_6H_5\\C_6H_5\\H\end{matrix}\right\}N,HCl$$

It is also formed by simply heating aniline hydrochloride to 300° C., (GIRARD and DE LAIRE), or by treating aniline with potassium (not sodium), and acting on the product with phenyl bromide (MERZ and WEITH).

Diphenylamine is, when pure, a solid crystalline body, melting at 45°, and distilling without change at 310° C. It is chiefly characterized by its power of yielding deep blue products of great tinctorial power on treatment with certain oxidizing agents (*Vide* BLUE DYES *infra*).

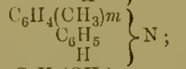
A great number of higher homologues of diphenylamine are known. Commercial diphenylamine is made from a mixture of aniline and toluidine (para- and meta-), not unfrequently containing higher homologues: hence it may be expected to contain, in addition to diphenylamine, the following bases:—

Paratoluyl phenylamine,



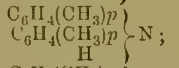
$$\left.\begin{matrix}C_6H_4(CH_3)p\\C_6H_5\\H\end{matrix}\right\}N;$$

Metatoluyl phenylamine,



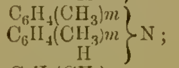
$$\left.\begin{matrix}C_6H_4(CH_3)m\\C_6H_5\\H\end{matrix}\right\}N;$$

Di paratoluylamine,



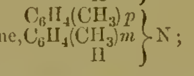
$$\left.\begin{matrix}C_6H_4(CH_3)p\\C_6H_4(CH_3)p\\H\end{matrix}\right\}N;$$

Di metatoluylamine,



$$\left.\begin{matrix}C_6H_4(CH_3)m\\C_6H_4(CH_3)m\\H\end{matrix}\right\}N;$$

Paratoluyl metatoluylamine,

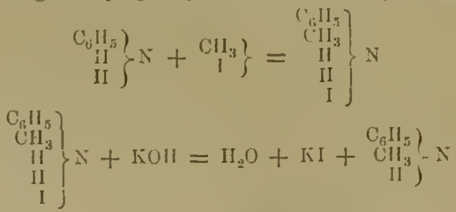


$$\left.\begin{matrix}C_6H_4(CH_3)p\\C_6H_4(CH_3)m\\H\end{matrix}\right\}N;$$

Besides higher homologues. The existence of all these bodies, however, has not yet been absolutely proved; some of them have been described, but it is not yet proved that the bodies examined were not mixtures of isomerides.

Methyl diphenylamine, $\left.\begin{matrix}C_6H_5\\C_6H_5\\CH_3\end{matrix}\right\}$ N, has been obtained by BARDY by the action of methylic alcohol on diphenylamine under pressure; by the action of oxidizing agents it forms blue and violet colouring matters.

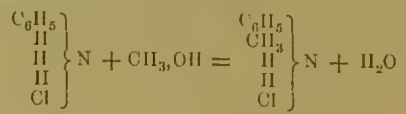
Methyl aniline, $\left.\begin{matrix}C_6H_5\\CH_3\\H\end{matrix}\right\}$ N.—This base is obtainable by the action of methyl chloride, bromide, or iodide on aniline, and the action of caustic alkalies on the resulting methyl phenylium salt formed (HOFMANN).



$$\left.\begin{matrix}C_6H_5\\H\\H\end{matrix}\right\}N + \left.\begin{matrix}CH_3\\I\end{matrix}\right\} = \left.\begin{matrix}C_6H_5\\CH_3\\H\\H\\I\end{matrix}\right\}N$$

$$\left.\begin{matrix}C_6H_5\\CH_3\\H\\H\\I\end{matrix}\right\}N + KOH = H_2O + KI + \left.\begin{matrix}C_6H_5\\CH_3\\H\end{matrix}\right\}N$$

Methyl nitrate, also, forms much methyl aniline on heating to 100° C. with aniline; its use, however, requires caution, as dangerous accidents and explosions have occurred through the volatility of the nitrate and its detonating power, under conditions not thoroughly understood. It is producible much more cheaply by the reaction of methylic alcohol on aniline hydrochloride under pressure (BARDY, POIRRIER, and CHAPPAT), the reaction being

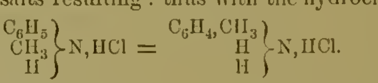


$$\left.\begin{matrix}C_6H_5\\H\\H\\H\\Cl\end{matrix}\right\}N + CH_3,OH = \left.\begin{matrix}C_6H_5\\CH_3\\H\\H\\Cl\end{matrix}\right\}N + H_2O$$

Methyl aniline hydrochloride is thus formed, from which the free base is obtainable by treatment with alkalies. Simultaneously with methyl aniline, dimethyl aniline hydrochloride is usually produced in variable quantities. It seems not improbable that this reaction is really due to the formation of a small quantity of methyl chloride which then attacks the aniline present, in accordance with HOFMANN's reaction; instead of aniline hydrochloride a mixture of aniline and sal ammoniac may be used, or a mixture of aniline and methylamine hydrochloride. When pure, methyl aniline is an oily liquid, boiling at 192° C., yielding colouring matters in small quantities only by the action of oxidizing agents; when mixed with dimethyl aniline, or with higher homologues, *e.g.*, methyl toluidine, the yield is much greater.

Dimethyl aniline, $\left.\begin{matrix}C_6H_5\\CH_3\\CH_3\end{matrix}\right\}$ N, is obtainable from methyl aniline by the same processes as those by which the latter is producible from aniline: it is a liquid, boiling at 202° C. (LAUTH), at 192° C. when perfectly pure (HOFMANN).

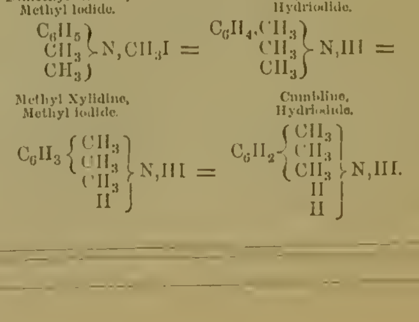
When the salts of methyl aniline are heated in a sealed tube to 350° C. for a day, they undergo a change of the kind referred to as "change of position," toluidine salts resulting: thus with the hydrochloride



$$\left.\begin{matrix}C_6H_5\\CH_3\\H\end{matrix}\right\}N,HCl = \left.\begin{matrix}C_6H_4,CH_3\\H\\H\end{matrix}\right\}N,HCl.$$

It is especially noteworthy that whilst paratoluidine (solid) results in the case of methyl aniline hydrochloride, a liquid toluidine, apparently metatoluidine, is formed when methyl aniline hydriodide is thus treated (HOFMANN).

Similarly, when the methyl iodide of dimethyl aniline (trimethyl phenylium iodide) is heated to a high temperature in a sealed vessel, or if dimethyl aniline hydrochloride is heated to 300° C. for ten hours with a large excess of methylic alcohol, analogous changes result; true homologues of aniline, or derivatives thereof, being formed by analogous reactions of "change of position." Thus with dimethyl aniline methyl iodide—



Dimethyl Aniline, Methyl Iodide. — Dimethyl Toluidine, Hydriodide.

$$\left.\begin{matrix}C_6H_5\\CH_3\\CH_3\end{matrix}\right\}N,CH_3I = \left.\begin{matrix}C_6H_4,CH_3\\CH_3\\CH_3\end{matrix}\right\}N,HI =$$

Methyl Xylidine, Methyl Iodide. — Cumidine, Hydriodide.

$$C_6H_3\left\{\begin{matrix}CH_3\\CH_3\\CH_3\\H\end{matrix}\right\}N,HI = C_6H_2\left\{\begin{matrix}CH_3\\CH_3\\CH_3\\H\\H\end{matrix}\right\}N,HI.$$

When the methylation of the resulting products is pushed further, as when the heating is effected in contact with excess of methylic alcohol, yet higher homologues are formed: thus there are formed successively derivatives of:—

Dimethyl aniline, $\left.\begin{matrix}C_6H_5\\(CH_3)_2\end{matrix}\right\}N$;

Dimethyl toluidine, $\left.\begin{matrix}C_6H_4, CH_3\\(CH_3)_2\end{matrix}\right\}N$;

Dimethyl xylidine, $\left.\begin{matrix}C_6H_3(CH_3)_2\\(CH_{3\ 2})\end{matrix}\right\}N$;

Dimethyl cumidine, $\left.\begin{matrix}C_6H_2\,(CH_3)_3\\(CH_3)_2\end{matrix}\right\}N$;

Dimethyl cymidine, $\left.\begin{matrix}C_6H(CH_3)_4\\(CH_3)_2\end{matrix}\right\}N$.

—HOFMANN and MARTIUS.

Amongst the products formed by the carrying out of this action to its limit is a hydrocarbon, apparently $C_6(CH_3)_6$; whilst a primary amine, $\left.\begin{matrix}C_6(CH_3)_5\\H_2\end{matrix}\right\}N$, has been obtained in small quantity by HOFMANN amongst the bye products of the action of heat on trimethyl phenylium iodide.

The occurrence of these reactions has doubtless a most important bearing on the colour-producing properties of methyl aniline and dimethyl aniline. HOFMANN's recent researches tend to show that the violet dye obtained by the oxidation of dimethyl aniline is *identical* with that got by methylating rosaniline, produced by oxidizing a *mixture of aniline and paratoluidine.*

Ethyl aniline, $\left.\begin{matrix}C_6H_5\\C_2H_5\\H\end{matrix}\right\}N$.—This base is obtainable from aniline and ethyl iodide, &c., or from aniline hydrochloride and ethyl alcohol, in the same way as methyl aniline is prepared: it is a colourless liquid, boiling at 204° C. Notwithstanding the close connection between the dyes got by oxidizing methylated anilines and those by methylating rosaniline, ethyl aniline does not yield by oxidation any noteworthy quantity of dyes such as those got by ethylating rosaniline. The following bodies:—

Diethyl aniline, $\left.\begin{matrix}C_6H_5\\C_2H_5\\C_2H_5\end{matrix}\right\}N$;

Amyl aniline, $\left.\begin{matrix}C_6H_5\\C_5H_{11}\\H\end{matrix}\right\}N$;

Methyl toluidine, $\left.\begin{matrix}C_6H_4CH_3\\CH_3\\H\end{matrix}\right\}N$;

Ethyl toluidine, $\left.\begin{matrix}C_6H_4CH_3\\C_2H_5\\H\end{matrix}\right\}N$;

Diamyl aniline, $\left.\begin{matrix}C_6H_5\\C_5H_{11}\\C_5H_{11}\end{matrix}\right\}N$;

Dimethyl toluidine, $\left.\begin{matrix}C_6H_4,CH_3\\CH_3\\CH_3\end{matrix}\right\}N$;

and hosts of analogous substances are known, and attempts have been made to utilize some of them as colour-producing agents, but for the most part with little or no success. Methyl toluidine and dimethyl toluidine (both para- and meta-) probably occur in the mixture of methyl aniline and dimethyl aniline, which is now largely manufactured for the production of "Violet de Paris;" but they do not contribute to the improvement of the dye, and it is a great object to obtain an aniline free from higher homologues for this particular purpose. Aniline containing more than 5 per cent. of toluidine is unsuitable for the production of Violet de Paris (LAUTH).

Chloraniline, $\left.\begin{matrix}C_6H_4Cl\\H_2\end{matrix}\right\}N$, and its homologues have been proposed as sources of colouring matters by POULAIN. When a mixture of chloraniline and chlorotoluidine is heated, hydrochloric acid and rosaniline are formed. The chlorinated bases are obtained, in the first instance, by acting on benzene with iodine and chlorine, when chlorobenzene is formed; this is then nitrated and converted into a chlorinated aniline oil in the usual way (*Vide article* BENZOL). By acting on these chlorinated bases with iodide of methyl, ethyl, &c., *methyl chloraniline*, $\left.\begin{matrix}C_6H_2Cl\\CH_3\\H\end{matrix}\right\}N$, and analogous products are formed; and by the withdrawal of the elements of hydrochloric acid from these substances, or mixtures of them, other dyestuffs can be produced.

MANUFACTURE OF ANILINE OILS.

The various kinds of aniline oils (including in this term the commercial bodies sold as aniline, toluidine, xylidine, and their mixtures) are all prepared in the same way, the sole difference being in the nature of the nitrobenzol used, *i.e.*, in the character of the hydrocarbon or "benzol" used in the first instance.

The method first tried on the large scale (by Messrs. SIMPSON, MAULE, and NICHOLSON) was that of ZININ, viz., the action of sulphuretted hydrogen on the solution of nitrobenzol in ammoniacal alcohol. The alcoholic solution, placed in a digester furnished with a condensing worm, was first saturated with ammonia gas, and then sulphuretted hydrogen was led in to saturation; the temperature was then raised to 100° C. by a steam worm, the taps of the digester being closed, so that the pressure was considerable. In a few hours all odour of sulphuretted hydrogen disappeared, that of ammonia only remaining; the liquid was then cooled, and again saturated with sulphuretted hydrogen and heated to 100° C. When the reaction was completed (which was known by the perfect solubility in dilute hydrochloric acid of a small sample drawn from time to time as a test), the ammoniacal alcohol was distilled off and used over again, the aniline remaining in the still, mixed with sulphur from the decomposition of the sulphuretted hydrogen; thus, in the case of aniline:—

$$\overset{\text{Nitrobenzene.}}{C_6H_5,NO_2} + 3H_2S = \overset{\text{Aniline.}}{C_6H_5,NH_2} + 2H_2O + 3S$$

and similarly for the higher homologues.

The inconvenience of this method and its comparative costliness soon led to its abandonment; the same may also be said of the process invented by HOFMANN and worked for a time by E. KOPP, viz., the addition to nitrobenzol (contained in a still) of zinc (in lumps), and the gradual addition of slightly diluted hydrochloric acid, an agitator being kept in motion to promote the reaction; when the reduction was complete chalk was added to decompose the aniline salts formed, and the contents of the still submitted to distillation, a dull red heat being finally applied. The processes proposed by Kremer (action of 2 to 2½ parts of zinc dust, and 5 of water, on 1 of nitrobenzol, the action going on spontaneously when started by heating): Vohl (action of glucose and alkalies upon nitrobenzol); and Wöhler (action of alkaline arsenites on nitrobenzol)—never came into very general use; the process of Béchamp (the one now universally adopted) being much preferable for commercial purposes. The original mode of working was as follows (Girard and De Laire):—Into cast-iron vessels 1 mètre high, 1·5 diameter, and 4 centimètres in thickness there is introduced a mixture of—

Iron filings,	200 kilogrammes.
Nitrobenzol,	100 "
Acetic acid at 40°,	100 "

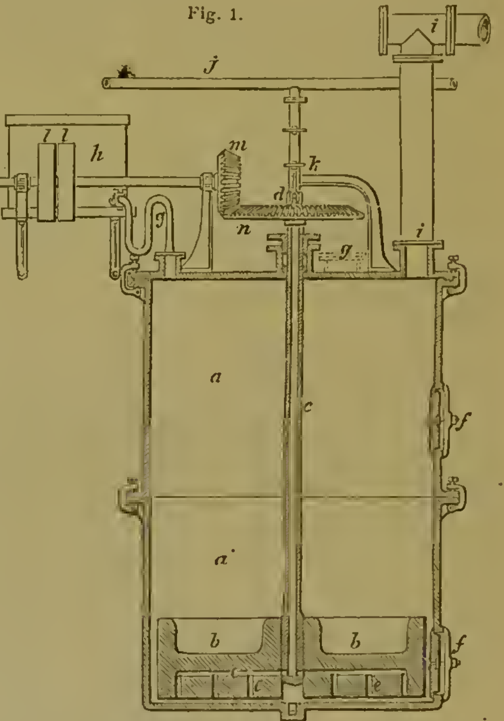
which fills the vessels about three-quarters full. These pots are covered by a dome furnished with a condenser, so that any products volatilized during the reaction may be recovered; an agitator moved by hand is provided to mix the ingredients. The iron being first introduced, the acetic acid is run in, and about a quarter of the nitrobenzol; in a few minutes a vigorous reaction begins and much vapour is evolved; this is partially condensed by the application of wet cloths to the dome, partly in the condensing worm. The rest of the nitrobenzol is similarly added from time to time as the reaction slackens; finally the whole is well mixed by means of the agitator, and a gentle heat applied to complete the reaction. On cooling a pasty mass results, which is removed and mixed with 10 to 12 per cent. of slacked lime; this mixture is then distilled in small retorts holding about 100 litres each, and provided with hand agitators to keep the mass from overheating at the sides. Aniline then passes over, its vaporization being much facilitated by the presence of acetone vapour, and the permanent gases arising from the decomposition of the calcium and ferrous acetates present.

This mode of operation presents several inconveniences; besides cost of fuel and labour, the aniline is largely contaminated with bodies of high boiling point (azobenzene, paraniline, xenylamine, acetanilide, &c.), either formed along with the aniline, or produced by its partial decomposition by the heat employed towards the end of the distillation; hence considerable modifications have been made in the process.

The following diagram (Fig. 1) illustrates the form of apparatus recommended by Girard and De Laire ("Traité des Derivés de la Houille," Paris, 1873); the cylinder in which the reaction is carried out is made of dimensions suitable to the charges worked—for batches of 500 kilos. of nitrobenzol, it is made one mètre in diameter, and two in height. It is provided with two condensing worms, one so arranged that the condensed liquid drops back into the vessel (cohobating worm), one arranged as in ordinary distillation (condensing worm). The materials are agitated by steam power, the shaft of the agitator being hollow and connected with a steam pipe; the vanes of the agitator are likewise hollow and perforated with holes, in such a way that jets of steam can be injected into the vessel through the agitator itself.

The cast-iron cylinder is in two parts, *a* and *a′*, which are united together by flanges and screw-bolts; *b*, *b*, is the agitator; *c*, the hollow axle of agitator; *d*, a pipe leading steam from the steam-pipe, *j*, to the agitator; *e*, *e*, perforations in agitator through which the steam issues; *f*, *f*, manholes; *g*, *g′*, tubulations for introduction of the materials; *h*, reservoir of nitrobenzol communicating with the cylinder by a syphon pipe; *i*, exit tube for vapours, communicating with the cohobating and condensing worms, either of which can be brought into connection with the cylinder or disjoined from it by opening or closing a cock; *k*, screw-bolt arrangement connecting steam-pipe with hollow axle; *l*, *l*, pulleys; and *m*, *n*, cog-wheels communicating motion to the agitator.

Fig. 1.



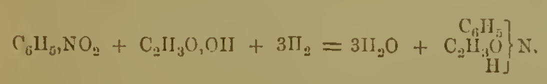
The whole of the nitrobenzol is first run into the cylinder together with the water and acid, and the iron filings are then added in batches of 50 kilos. each; a vigorous reaction ensues, and much vapour is evolved, which is condensed by the cohobating worm (the condensing worm being shut off); after the lapse of half an hour another batch of iron is introduced, and so on until the whole of the nitrobenzol is reduced, which is known by the perfect solubility in dilute hydrochloric acid of a sample drawn for the purpose. The cohobating worm is then shut off and the condensing worm connected; the slacked lime is then added through the tubulation for the purpose (*g*), and superheated steam (at 5 to 6 atmospheres) blown in through the agitator; the heat effects the reduction of the last traces of nitrobenzol, and when the cylinder is sufficiently hot, aniline and water are condensed in the worm. The former being only sparingly soluble in water is readily separable from the aqueous distillate; the small quantity of aniline dissolved in the latter is recovered by adding salt to the liquor, from which the aniline then separates, being much less soluble in brine than in plain water. The condensed water may also be used for generating steam for a second operation, whereby the dissolved aniline is also recovered. BOLLEY recommends the steam supply to be so managed, that about 14 parts of water condense for every 1 of aniline.

Various minor modifications in the shape and size of the aniline still, the kind of agitator used, &c., &c., have been adopted by different manufacturers. Thus many manufacturers dispense with a cohobating worm, using simply a still provided with an agitator through which steam can be injected at the close of the operation; the distillate which passes over during the first energetic action of the iron on the nitrobenzol is returned to the still, as it contains a good deal of unaltered nitrobenzol, which has been carried over with the steam, &c.

The most important variations in BÉCHAMP's process as worked by different aniline makers lie in the relation between the quantities employed of nitrobenzol, acetic acid, and iron; if too much of the latter substance be used the reaction is apt to go too far, more hydrogen being added on than is required to form aniline, and ammonia and benzol being formed instead; thus with nitrobenzene itself;—

$$C_6H_5,NO_2 + 3H_2 = C_6H_5,NH_2 + 2H_2O$$
$$C_6H_5,NO_2 + 4H_2 = C_6H_6 + NH_3 + 2H_2O$$

and so on for the higher homologues. For this reason it is preferable to conduct the reducing process as above described (*i.e.*, to add the iron gradually to the whole of the nitrobenzol), rather than to do as some manufacturers do, viz., put all the iron into the still, and then add the nitrobenzol in small portions at a time. In the latter way of working, the nitrobenzol is always in contact with excess of iron. The presence of too much acetic acid gives rise to the production of acetanilide and its homologues,



$$C_6H_5,NO_2 + C_2H_3O,OH + 3H_2 = 3H_2O + \left.\begin{matrix} C_6H_5 \\ C_2H_3O \\ H \end{matrix}\right\} N.$$

On the other hand, the presence of too little acetic acid and iron is favourable to the production of azobenzide from the deficiency in nascent hydrogen:—

$$2C_6H_5,NO_2 + 4H_2 = 4H_2O + C_{12}H_{10}N_2.$$

The most divergent statements are made by various writers as regards the proportions of iron filings, acetic acid, and nitrobenzol to be used in order to gain the best result; thus the following proportions have been given by the respective authorities named:

	Girard and De Laire (1873).	Bolley (1870).	Reimann (1867).	Kopp (1864).	Used by Renard and Franc in 1860. (Girard and De Laire).
Nitrobenzol,.....	100	100	100	100	100
Acetic acid,......	10- 2	5-10	50	90-100	100
Iron filings,......	140-140	200	150	140-150	200
Water,..........	50- 60	—	—	—	—
Hydrochloric acid,	10	—	—	—	—

The following proportions are those actually employed in some large aniline factories with good results:—

(A)		(B)	
Nitrobenzol,.........	100	Nitrobenzol, ...	13
Acetic acid at 8° B (sp. gr. 1·06)	8·	Acetic acid,..........	6
Iron,..................	144	Iron filings,..........	15
		Water,..............	4

Of these proportions A corresponds more nearly with those given by GIRARD and DE LAIRE (1873), and B with those given by REIMANN (1867).

The yield of aniline oil necessarily varies with the nature of the nitrobenzol used; pure nitrobenzene should theoretically yield 75·6 per cent. of aniline; pure nitrotoluene, 78·1 per cent. of toluidine. In practice somewhat less than the theoretical amount is obtained, owing to the formation of bye products and to the presence of impurities in the nitrobenzol, &c.; something like 66 parts of aniline oil per 100 of nitrobenzol is a fair average yield. KREMER's process (see *ante*) yields about 65 parts from 100 of good nitrobenzol, if properly conducted.

It was formerly supposed that the reduction of nitrobenzol to aniline occurred in virtue of the reaction—

$$C_6H_5,NO_2 + 2\,Fe + H_2O = Fe_2O_3 + C_6H_5,NH_2$$

peroxide of iron being formed; but SPILLER has recently shown (British Association Reports, 1873) that magnetic oxide of iron is produced, thus:—

$$4C_6H_5,NO_2 + 9\,Fe + 4H_2O = 3F_3O_4 + 4C_6H_5,NH_2$$

The crude aniline oil is next subjected to a process of fractional distillation; the lowest portions of distillate frequently contain benzene and its homologues, acetone, odorine, &c., &c. Usually the oils are purified by the colour maker himself, that he may obtain the particular mixtures he prefers. In some cases, however, the oils are prepared for the market according to contracts, specifying the proportion of the substance that shall distil between certain fixed limits. Light aniline oils distil for the most part below, and heavy aniline oils chiefly above 200° C. The term *queues d'aniline* (aniline tailings) is applied to the highest boiling heavy oils.

REIMANN distinguishes the light and heavy oils by the names *kuphaniline* and *baraniline* respectively, and gives the following tables as to the composition of various kinds of commercial oils:—

	Kuphaniline.	Baraniline.
	Per cent.	Per cent.
Water, odorine, &c.,...........	5	—
Aniline,.......................	90	—
Toluidine (para- and meta-),.....	5	70
Higher homologues,............	—	30
	100	100

Distinguishing these by the letters K and B respectively, the following results are found, as the result of carefully conducted experiments made by that chemist:—

Distilling below Degrees C.	Mixtures Containing—										
	K = 100 B = 0	K = 90 B = 10	K = 85 B = 15	K = 80 B = 20	K = 75 B = 25	K = 62·5 B = 37·5	K = 60 B = 40	K = 50 B = 50	K = 37·5 B = 62·5	K = 25 B = 75	K = 0 B = 100
180°	2·5	7	2·5	5·5	3·5	4	—	4	2	3	—
—	6	—	—	22	3·5	3	—	3	2	2·5	—
185°	51	50	29·5	—	5·5	2·5	7	4·5	—	2·5	—
—	—	—	—	—	—	—	—	—	—	—	2
190°	34	34	56·5	55·5	55·5	41	37	7·5	5·5	4·5	1 5
195°	—	5	7·5	8·5	15	25	33	42	40	17	8
200°	—	—	—	—	9	8·5	—	19	28·5	36	18
205°	—	—	—	—	4·5	5	16	10	11	16	39
210°	—	—	—	—	—	4·5	—	3·5	7·5	8	19
215°	—	—	—	—	—	—	—	—	—	4·5	7
Residue,	3·5	4	4	8·5	3·5	6·5	7	6·5	3·5	5	5·5

The kind of oil most suitable for making fuchsine is that containing 75 per cent. of kuphaniline and 25 baraniline, *i.e.*, of which 92 per cent. distils below 200° and 68° below 190°.

The following apparatus is described by Girard and De Laire for the fractional distillation of crude aniline oils ("Traité des Dérivés de la Houille," Paris, 1873) (Aniline, Plate 1.):—A distilling vessel provided with two necks, and having a capacity of about a cubic mètre, is heated by a fire; the aniline vapour issuing from the neck is made to traverse a series of condensing tubes immersed in a tank of aniline oil, so arranged that what is condensed in these worms returns to the still: the lowest boiling substance, not being wholly condensed, passes on to another condensing worm, and is thus separated. The aniline oil in the tank becomes heated and begins to distil also, the tank being provided with a separate condensing worm.

In the accompanying engraving (Aniline, Plate 1.) *a c* is the neck of the still communicating with the sinuous condensing tube *k*; *b e*, a second neck, for reflux of liquid condensed in *k*; *d d*, a tube connecting the tank *i* with its condenser *o*; *f f*, a tube connecting the sinuous tube *k*, with its condenser *n*; *g*, a two-necked still; *h*, manhole; *i*, a tank for the second batch of aniline, to be distilled by means of the heat given out by the condensation of vapours in the sinuous tube *k*; *j*, the reflux tube; *l*, tap for emptying still; *m m*, thermometers; *n*, condensing worm for vapour from the still *g*; *o*, condensing worm for vapour from tank *i*.

By means of an arrangement such as the foregoing it is easy to separate a crude oil into fractions possessing a composition approaching to the following:—

LIGHT ANILINE OILS.

Percentage,..........	3·7	34·2	53·7	7·1	1·3
Distilling at °C,....	below 180°	180° to 185°	185° to 190°	190° to 195°	above 195°

HEAVY ANILINE OILS.

Percentage,..........	10	20	40	22	8
Distilling at °C,....	below 195°	195° to 200°	200° to 205°	205° to 210°	above 210°

Aniline Tailings.

Very little aniline, a little toluidine, xylidine, cumidine, &c., together with bye products such as phenylene diamine, paraniline, xenylamine, azobenzide, acetaniline, and other homologues, &c.

Krouber gives the following valuable tables illustrating the general characters of the benzols, nitrobenzols, anilines, and fuchsines, derivable the one from the other:—

	Boiling point of Benzol.	Sp. gr. of Benzol at 15°.	Principal boiling point of Nitrobenzol.	Sp. gr. of Nitrobenzol at 16 .	Yield of Aniline Oil per 100 parts of Nitrobenzol.	Principal boiling point of Aniline Oil.	Sp. gr. of Aniline Oil at 16°.	Yield of Colour obtainable, Crystallizable Fuchsine being taken as 1000.	Tint of Colour communicated to Goods dyed therewith.	
	Degrees C.		Degrees C.			Degrees C.				
a	83–84	0·9118	205–210	1·1591	50	180–185	1·0205	5	Dirty violet.	Contains chiefly violaniline.
b	80–85	0·9263	205–210	1·1617	55	180–185	1·0199	20	Reddish violet.	
c	85–90	0·9154	210–215	1·1577	56	185–190	1·0181	110	Violet red....	Mauvaniline with a little rosaniline.
d	90–95	0·9210	210–215	1·1445	63	185–190	1·0139	160	Red.	
e	95–100	0·9089	215–220	1·1425	66	190–195	1·0109	230	Red.	
f	100–105	0·9071	220–225	1·1365	73	195–200	1·0060	270	Red.	
g	105–110	0·9048	220–225	1·1319	74	195–200	1·0018	240	Red.	
h	110–115	0·9033	225–230	1·1235	69	200–205	1·0009	260	Red..........	Contains much chrysotoluidine.
i	115–120	0·9022	225–230	1·1187	74	200–205	0·9975	260	Yellowish red..	
j	120–125	0·9009	230–235	1·1182	73	205–210	0·9943	200	Red.	
k	125–130	0·9001	230–235	1·1093	74	205–210	0·9926	180	Red.	

The benzol marked *a* was crystallizable. The nitrobenzols and anilines derived from these benzols yielded the following quantities of distillates at the various temperatures indicated:—

Nitrobenzol from Benzol marked	Range of Temperatures. Degrees C.												Total Distillate.
	195° 200°	200° 205°	205° 210°	210° 215°	215° 220°	220° 225°	225° 230°	230° 235°	235° 240°	240° 245°	245° 250°	250° 255°	
a	2	3	93	2	—	—	—	—	—	—	—	—	100
b	—	—	52	40	7	1	—	—	—	—	—	—	100
c	—	—	11	64	13	9	3	—	—	—	—	—	100
d	—	3	5	52	32	7	1	—	—	—	—	—	100
e	—	2	2	11	58	15	11	1	—	—	—	—	100
f	—	—	3	4	28	43	16	5	1	—	—	—	100
g	—	—	1	3	4	48	31	11	2	—	—	—	100
h	—	—	1	3	4	18	51	18	4	1	—	—	100
i	—	—	—	2	2	6	41	34	11	4	—	—	100
j	—	—	—	2	2	6	24	40	13	9	4	—	100
k	—	—	—	1	3	3	10	37	29	13	3	1	100

Aniline from Benzol marked	Range of Temperatures. Degrees C.										Total Distillate.
	Below 180°	180° 185°	185° 190°	190° 195°	195° 200°	200° 205°	205° 210°	210° 215°	215° 220°	220° 225°	
a	5	92	3	—	—	—	—	—	—	—	100
b	4	78	14	4	—	—	—	—	—	—	100
c	3	28	61	8	—	—	—	—	—	—	100
d	—	5	60	29	6	—	—	—	—	—	100
e	—	4	9	64	16	7	—	—	—	—	100
f	—	—	4	38	46	8	4	—	—	—	100
g	—	—	—	5	54	29	8	4	—	—	100
h	—	—	—	4	32	53	7	4	—	—	100
i	—	—	—	—	5	62	24	6	3	—	100
j	—	—	—	—	4	25	50	15	6	—	100
k	—	—	—	—	—	6	52	29	8	5	100

With various mixtures of the foregoing benzols, &c., KROUBER obtained the following results:—

Mixtures of Aniline from Benzols marked (equal quantities of each kind of Aniline)	Boiling points of Benzols used.	Relative quantities of Colour produced.	Shades of Colour communicated to Goods dyed therewith.
	Degrees C.		
b + *c*	80–90	16	Very violet red (mauvaniline and rosaniline).
d + *e*	90–100	17	Red.
f + *g*	100–110	32	Red.
h + *i*	110–120	24	Yellowish red (rosaniline and chrysotoluidine).
j + *k*	120–130	18	Red.
b + *c* + *d* + *e*	80–100	24	Violet red.
d + *e* + *f* + *g*	90–110	37	Red.
f + *g* + *h* + *i*	100–120	31	Red.
h + *i* + *j* + *k*	110–130	19	Red.
b + *c* + *d* + *e* + *f* + *g* + *h* + *i* + *j* + *k*	80–130	25	Red.

The best results, therefore, are obtained when a benzol boiling principally between 90° and 100° C. is used, giving an aniline boiling for the most part between 185° and 205° C. This result differs but slightly from REIMANN'S (see *ante*), the best oil for fuchsine being stated by him to boil 61 per cent. between 185° and 190° C., and 28·5 per cent. between 190° and 205° C. GIRARD and DE LAIRE state that the most suitable oil is that composed of sensibly equal quantities of true aniline and toluidine, and boiling 10 per cent. between 182° and 185° C., 40 per cent. between 185° and 190° C., 40 per cent. between 190° and 195° C., and 10 per cent. between 195° and 200° C. KOPP states that the best fuchsine oils are found to boil almost wholly between 185° and 210° C.; whilst HOFMANN found that various specimens of the oils actually in use on the large scale boiled between 182° and 220° C.

MANUFACTURE OF DIPHENYLAMINE.

The apparatus employed by GIRARD and DE LAIRE is described by them as follows ("Traité des Derivés de la Houille," Paris, 1873):—

An autoclave of malleable iron 0·6 mètre diameter and 0·8 mètre in height is provided, the interior being enamelled. To the lid are affixed a safety valve, a metallic pressure gauge, and an iron tube closed at the bottom and dipping inwards in such a manner that the end of the tube is below the surface of the liquid in the autoclave; in this is placed a thermometer.* A condensing tube which can be shut off at pleasure by means of a cock is also adopted, so that the interior can be brought into free communication with the external air without the loss of volatile substances. The heat is communicated by a flue proceeding from a coke furnace, the draught of which is regulated by dampers; the direct action of the fire is thus avoided, only the hot products of combustion being used for heating the autoclave. The annexed diagram (Fig. 2,) illustrates the nature of the apparatus. A cohobator is there represented as also attached to the autoclave; this can be shut off at pleasure by means of a cock; it communicates with a cylindrical condensing vessel immersed in water, the purpose of which is to regain ammonia liberated during the reaction: 70 kilos. of perfectly dry aniline hydrochloride, and 50 of aniline are introduced into the autoclave and the fire raised; the thermometer quickly mounts up to 200° C., the pressure gauge indicating no excess of pressure. After this point is reached the thermometer rises only slowly, an increased pressure being noticed if all the taps are closed. It is found by experience that if the ammonia generated in the reaction be allowed to remain in the digester, the yield of diphenylamine is much less than if the ammonia be allowed to escape; in fact Girard and De Laire have found the inverse reaction to take place when a salt of ammonia, and one of diphenylamine, are heated together. In order to avoid this, the cohobator is opened so that the evolved ammonia may pass off into its appropriate condenser, the aniline being condensed and falling back into the digester. After about two hours the cohobator is shut off, the thermometer indicating a temperature of 215° or 220° C.; the heat being continued, the temperature gradually rises to 250° C., whilst the pressure mounts up to 5 atmospheres. The whole operation lasts about twelve hours, from 60 to 77 per cent. of diphenylamine being obtained; if, however, the whole of the ammonia be retained in the autoclave, the yield does not exceed from 20 to 30 per cent.

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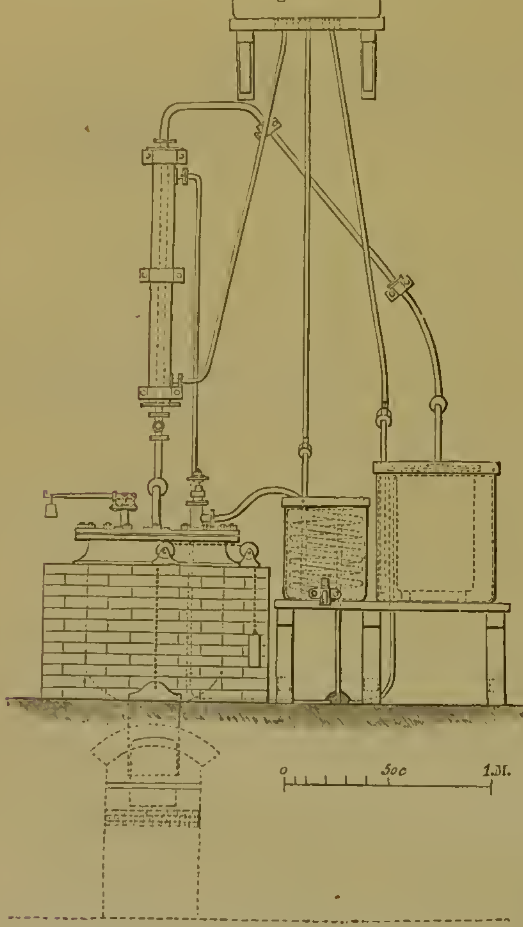
* The indications of the thermometer are more accurate, and the fluctuations of temperature are registered less slowly, if the tube is filled to the height of some inches with mercury.

Fig. 2.



The diphenylamine is separated from unaltered aniline by adding to the cooled product 70 kilos. of hydrochloric acid (which dissolves the whole), and next pouring it into 300 to 400 litres of water; diphenylamine then separates and falls to the bottom, whilst aniline hydrochloride remains dissolved. The precipitate is washed with a little boiling water and finally with an alkaline ley to decompose any residual diphenylamine hydrochloride, and is then either distilled in retorts with a large heating surface, or is recrystallized from petroleum oils. The aniline hydrochloride dissolved in the aqueous liquors is regained by evaporation, &c., and used over again.

The commercial diphenylamine thus obtained usually contains a considerable admixture of the higher homologues. A mixture of about 18 parts of diphenylamine and 11 of ditoluylamine produces a pure deep blue on oxidation by carbon sesquichloride.

MANUFACTURE OF METHYL ANILINE AND DIMETHYL ANILINE.

The process at first employed for the production of this body necessitated the use of iodide or bromide of methyl, and hence was far too costly to permit of any large development of the industry. Into an enamelled autoclave aniline is introduced, and then in successive portions the requisite quantity of methyl salt; much heat being generated during the mixture, it is necessary to add the methyl salt in such a way as to avoid any danger of mechanical projection. This is effected by employing a reservoir consisting of a tube with an expansion thereon, with a tap above and below the vessel thus formed; this is adapted to the lid of the autoclave. The top cock being open the methyl compound is poured in through a funnel; the top tap is then closed, and the lower one opened; the methyl salt thus flows gently into the autoclave, no projection being possible. This process is repeated until the requisite quantity of methyl salt is added; the autoclave is then heated to 100° C. for one or two hours. After cooling soda is added, and the liberated methyl aniline distilled off by steam.

Process of Bardy, Poirrier, and Chappat.—Fifty kilos. of pure aniline hydrochloride and 30 of methylic alcohol are introduced into an autoclave resembling that employed for the production of diphenylamine; the whole is then heated cautiously to between 250° and 300° C. for twelve hours. The reaction takes place for the most part at 280° C.; a sudden increase in pressure is frequently noticed, occasionally, it is stated, reaching 140 atmospheres. To avoid accidents, therefore, it is necessary to regulate the heat carefully, diminishing it greatly when the pressure begins to rise quickly, or even cooling the vessel. When the operation is completed, the product is treated with caustic soda ley, to decompose the hydrochlorides of methyl and dimethyl aniline formed; and these are then distilled off by steam, and rectified, if necessary, in an oil bath. Besides this mixture boiling near 200° C., other bodies of much higher boiling point are formed, such as dimethyl toluidine, dimethyl xylidine, &c. This mixture is used for Paris violet without any separation of the two constituents being attempted: the larger the quantity of methylic alcohol employed the greater is the quantity of dimethyl aniline present in the product, *cæteris paribus*.

POIRRIER and CHAPPAT state (Bull. Soc. Chim., Paris [2], vol. vi. p. 502) that by the action of heat on 100 parts of hydrochloride of aniline, and 50 to 80 parts of methylic alcohol, only three to four hours' treatment at 250° to 300° C. is required, the hydrochlorides being thus formed; whilst the action of 100 parts of aniline, 100 of sal ammoniac, and 50 to 80 of methylic alcohol at 300° C. requires several hours, and yields chiefly free methyl aniline. Nitric methyl ether (methyl nitrate) acts on aniline in the cold, but only slowly, producing methyl aniline; at 100° C. this action is rapid. Heating aniline with methylic alcoholic chloride (methyl chloride) at the ordinary pressure also forms methyl aniline. A larger or smaller quantity of dimethyl aniline is always formed simultaneously, according to the quantity of methyl compound used. These processes and corresponding ones for other substitution derivatives of aniline, are included in their patent (No. 71,970).

ANILINE DYES.

With the exception of mauveine, aniline black, and a few dyes not yet thoroughly investigated, all the aniline dyes in use are either derived from fuchsine or are closely connected with it. For convenience of reference, they may be grouped in accordance with their colour as follows:—

Red Dyes.—Fuchsine (also known under the name of Fuchsiacine, Chyraline, Azaleine, Erythrobenzene, Rosaniline, Pseudo-rosaniline, Aniline red, Magenta, Solferino, Roséine, Aniléine rouge, Fuchsinic acid, Rubine, &c., &c.); and several others of minor importance, such as Rouge de Toluene (Rosatoluidine, Toluene red), Rouge de Xylene (Xylidene red, Xyline red, Rosaxylidene), Chrysaniline red (Chrysotoluidine red), Geranosine, Saffranine, Furfurol red, Ulrich's scarlet, &c.

Blue Dyes.—Bleu de Lyons (Triphenyl rosaniline, Bleu de nuit, Bleu Lumiére, Bleuine, Girard and De Laire's blue, Night blue, Bleu de Paris, &c., &c.), Soluble blue (Nicholson's blue, Alkali blue), Diphenylamine blue; and several less well-known blues, such as Coupier's indigo blues, Rosatoluidine blues, Toluidine blue, Mulhouse blue, Azurine, Aldehyde blue, Azuline, Azodiphenyl blue, &c.

Violet Dyes and Dyes intermediate between Red and Blue.—Mauveine (Mauve, Aniline purple, Aniline violet, Perkin's violet, Aniléine, Phenameine, Violine, Rosolane, Tyraline, Harmaléine, Indisine, &c., &c.). Hofmann's violets, of various shades, Violet blue, Blue violet, Primula, &c., &c. Poirrier's and Chappat's violets or Violets de Paris (Methyl violets), of various shades; and several others of less importance, some of which are probably mixtures of reds and blues—Mono- and di- phenyl rosaniline (Violets de Lyons, Violet imperial, Dahlia, &c.), Regina purple (Nicholson's purple), Violaniline, Mauvaniline, Isopropyl violet, Turpentine violet (Britannia violet) Wise's violet, &c.

Green Dyes.—Iodine green or Hofmann's green, Methyl green (Vert de Paris, Vert soluble), Aldehyde green (Viridine, Aniline green), Perkin's green, Emeraldine. Mixed greens, usually mixtures of picric acid and blues or violets.

Miscellaneous Dyes.—Browns, yellows, oranges, greys, &c. Aniline black.

All the aniline dyes, with the exception of soluble blue (alkali blue), and a few analogous substances, are bases or compound ammonias. In many cases the base itself is colourless, its salts forming dyes of great intensity and beauty, such as rosaniline; what is used for dyeing, therefore, is uniformly a salt of the base. For some purposes the organic salts (the acetate, for instance), are preferred, for others salts of a mineral acid, *e.g.*, the hydrochloride. The nature of the tint communicated is usually only very slightly or not at all modified by varying the acid of the salt. Many of these bases in the free state are really hydrates (thus rosaniline = $C_{20}H_{19}N_3,H_2O$), the "water of hydration" being only lost by heating.

GENERAL HISTORY OF THE ANILINE DYES.

1. *Red Dyes. A. Fuchsine.*—In 1843 HOFMANN observed that a red product is formed when aniline is treated with fuming nitric acid; ZININ also made the same observation at about the same time. In 1856 NATANSON found that by acting on ethylene chloride with aniline at 200° C., a deep blood red colouring matter is formed; and at about the same time, HOFMANN obtained a similar substance as one of the products formed by heating to 170° to 180° C. for thirty hours, a mixture of carbon tetrachloride and aniline. There appears to be little doubt that NATANSON'S and HOFMANN'S products are identical with fuchsine. The manufacture of *Hofmann's red* has been more than once attempted on the commercial scale, and a modification of it is employed by GIRARD and DE LAIRE in the manufacture of diphenylamine blue. A committee of the Société de Mulhouse reported very favourably on the results of the process.

The technical use of red colours derived from aniline did not, however, commence till 1859, when VERGUIN formed a red dyestuff by the action of heat on a mixture of tin tetrachloride and commercial aniline oil. He immediately communicated his process to MM. RÉNARD FRÈRES and FRANC, of Lyons, who commenced manufacturing the dye forthwith, taking out a series of patents in France for the use of this and other metallic chlorides, and other substances. To this new colour the name *Fuchsine* was applied, according to one account in honour of the manufacturer (Rénard = Fuchs = Fox); according to another version, because of the resemblance in tint of silk dyed with this substance, to the flower of a variety of fuchsia plant (*Fuchsia coccinea*). Other chemists soon found that many substances of a dehydrogenizing character formed fuchsine when heated with aniline oil; but the French law courts decided that the use of such other substances constituted an invasion of patent right, and in consequence the manufacture of fuchsine in France became virtually a monopoly. The product of the most important of these new processes (that proposed by DURAND, SCHLUMBERGER, and also by GERBER-KELLER, viz., the action of nitrates, more especially mercuric nitrate, on aniline oils) was termed *Azaléine*. Although almost identical in properties with fuchsine, it is in fact dissimilar, the one being a nitrate of a compound ammonia, the other the corresponding hydrochloride. The corresponding acetate is sometimes designated *Roseine*, this name having been originally applied to a red dyestuff obtained by oxidation of aniline with sulphuric acid and peroxide of lead (see *infra*).

The number of processes for the conversion of aniline oils into a red dye (presumably identical with fuchsine, though frequently called by another name) is very large; some of these were obtained as bye products in the manufacture of mauve.

Thus Laurent and Casthelaz prepared a red dye, termed by them *Erythrobenzol*, by acting on nitrobenzol with iron filings and hydrochloric acid, and heating the mass. Probably the colouring matter was identical with fuchsine, being formed by a double reaction, viz.:—First, the action of the metallic chloride on the aniline, analogous to that of the tin tetrachloride; and secondly, the reaction of nitrobenzol on aniline, in which fuchsine is also formed. This latter process has been several times patented since by other chemists, and quite recently by Meister Lucius and Brüning, who are now working it on a large scale. Schlumberger proposed the use of corrosive sublimate and tin amalgam; Depouilly and Lauth utilized the reaction of aniline nitrate on aniline (several patents involving this reaction have been subsequently taken out); Blockley, Watson, and other chemists employed aqua regia in some form as an oxidizing agent; Gingon used gaseous chlorine; Mène nitric acid; Jacquemin ferric sulphate; Williams used mercuric phosphate or acetate; Dale and Caro a mixture of lead nitrate and phosphoric acid; Smith used antimonic anhydride; Gratrix a nitrate of antimony (*i.e.*, a mixture of antimonic oxide and nitric acid) or nickel nitrate; others heated aniline salts either *per se* or in contact with sand (probably the oxygen of the air was here the oxidizing agent). Wilson heated aniline with 5 per cent. of arsenic, nitric, or iodic acid to 120° C., and then gradually added manganese dioxide; whilst recently Ferriere has obtained a red dyestuff by the action on aniline acetate of an ammoniacal copper solution; and Claus and also Hamel have obtained a similar product by heating aniline with sulphur chloride.

All these processes, however, and others too numerous to mention in detail, have been practically found inferior for commercial purposes to the method now in general use, viz., that patented almost simultaneously, in England by Medlock and by Nicholson, and in France by Girard and De Laire; *i.e.*, the use of arsenic acid as a dehydrogenizing agent.

Medlock's patent was dated January 18, 1860; Nicholson's, January 26, 1860 (both in Great Britain); Girard and De Laire's was dated May 26, 1860 (France). Arsenic acid, however, had been previously mentioned as a fuchsine-producing agent in a patent specification of Gerber-Keller in 1859, and had been described by Béchamp in the latter end of 1850. Poulain patented in 1869 the indirect oxidation of aniline and its homologues by means of chlorination and heating.

Various preparations are met with in the trade, designated as cerise, maroon, &c., which are mainly impure fuchsine; for the most part these are extracted from residues and bye products of the manufacture.

B. *Rouge de toluene*, and C. *Xylidine red*, were introduced by Coupier in 1866, and are as yet but little known.

D. *Chrysaniline and chrysotoluidine red.*

E. *Oxidation and decomposition products of rosaniline, geranosine.* F. *Saffranine.* G. *Ulrichs' scarlet*, patented in 1869. H. *Rosolic acid.*

I. *Furfurol red.*—Stenhouse found in 1860 that when aniline acetate and furfurol are well mixed together at the ordinary temperature, a red colouring matter is formed, insoluble in water, but soluble in alcohol, wood spirit, and strong acetic acid. Ammonia dissolves it to a colourless liquor, which is reddened again by acids. It dyes silks and woollens of a fine red tint, but it is very unstable, and cannot resist the action of light, and even fades when kept in the dark. Attempts were made to manufacture it by Persoz, but without success. Toluidine forms a similar product (Stenhouse—Proc. Roy. Soc., xviii. p. 537).

2. *Blue Dyes.*—A. *Various.*—Fritsche found in 1840 that when aqueous aniline salts are treated with hydrochloric acid and potassium chlorate, indigo blue flakes are precipitated; in 1860, Calvert, Lowe, and Clift, utilized a similar reaction for the preparation of a blue dye, called by them *Azurine*, by the action of weak soda or soap solutions on goods printed with emeraldine.

Caro found that blue products were formed by the reaction of nitrous acid on fuchsine, and the partial reduction of the products; and Lauth observed in 1860 that blue bodies result from the action of hydrogen dioxide on aniline salts, or of certain reducing agents, such as stannous chloride, on rosaniline salts. These substances are probably closely allied to the two following dyestuffs.

Mulhouse Blue and Aldehyde Blue.—The former of these was obtained in 1861 by Gros Renard and Schaeffer by boiling together rosaniline nitrate, powdered shellac, carbonate of soda, and aqueous alcohol; the latter in 1860 by Lauth, being produced when an aldehyde, or a mixture of materials that will generate aldehyde, is made to act on a hot acid solution of aniline. The want of permanency of these dyes has led to their almost entire disuse; although, unlike most aniline colours, they possess the power of adhering to cotton without mordanting.

The fugitive blue, or violet blue, formed by the action of hypochlorites on aniline (Runge's reaction), and various blues of a more or less purplish cast, formed by various inventors who endeavoured to imitate Perkin's mauve, are probably related to these substances; their industrial importance is small, and of their chemical composition nothing whatever is known, save that Runge's blue is closely allied to mauveine, as it gives rise to that substance on boiling with alcohol (Perkin). Guignon Marnas and Bonnet have prepared a blue aniline derivative, *Azuline*, by heating coralline (*q. v.*) with aniline.

B. *Phenyl and Tolyl Substitution Derivatives of Fuchsine.*—Soon after the publication of the arsenic acid process for making fuchsine, it was noticed that by the use of an insufficient quantity of acid (less than one equivalent for one of aniline) violet and even blue products were obtained instead of red. Thus, in 1861, Girard and De Laire patented the use of a mixture of 1 part acid and 1½ to 2 parts aniline for the production of violet and violet blue dyestuffs; in a later patent the action of aniline on a ready formed rosaniline salt was specified for the same purpose, the first product being a violet dye, which by further treatment gives a blue product. Similar patents were subsequently taken out by many chemists, in order to overcome certain practical difficulties in the way of obtaining a pure blue, which could not be obtained by the patented process of Girard and De Laire, although every shade of violet could be readily obtained by that method. To effect this it is essential that either an *organic* rosaniline salt should be used, or else that an *organic* potassium or sodium salt should be also added. Thus Monnet and Dury, and also Nicholson, treated a mixture of rosaniline acetate (formed by adding free rosaniline to acetic acid) with aniline. Gilbee heated rosaniline with aniline acetate. Price proposed to heat fuchsine to 159° to 190° C. with aniline acetate, valerate, lactate, benzoate, tartrate, or oxalate, until the required shade is attained. Passavant heated magenta with aniline and acetate of soda; the potassium salt gives a slight shade somewhat inclining to greenish, according to Price, and also Levinstein. Wanklyn, Bolley, and also Holliday, employed benzoic acid in the manufacture of aniline blue; C. G. Williams, oleate or oxalate of aniline. Schlumberger treated a mixture of fuchsine with aniline acetate and as much sodium carbonate as would just decompose the latter, and so on.

In working the process, many manufacturers heat a mixture of fuchsine, aniline, and sodium acetate until the colour becomes a blue violet or reddish blue, and then add to the mass benzoic acid, benzoate of potassium or sodium, sodium formate, stearic acid, sodium stearate, or common tallow soap, in small quantities at a time, until the proper shade appears; of these, soap is the substance most frequently employed.

The crude product obtained in the manufacture of aniline blue is (when freed from excess of aniline) frequently sold under the name of *Direct blue;* this again, when purified in various ways, is sold as *Purified blue* and *Night blue* (*Bleu Lumière*), the latter name being applied to such special varieties as communicate to goods dyed with them the power of retaining a pure blue tint by artificial light. This blue has been known by a variety of names, more especially as *Bleu de Lyons*, *Triphenyl rosaniline blue*, *Girard and De Laire's blue*, and *Aniline blue;* the term *Paris blue* (Bleu de Paris) has also been applied to it; but as this same term has been applied successively to nearly every other blue or violet blue invented, and is, moreover, often used on the Continent to indicate Prussian blue (ferric ferrocyanide), it is a name not to be recommended. A dye very similar to, if not identical with Bleu de Lyons, was also produced in 1866 by Girard and De Laire by oxydizing commercial diphenylamine, containing diparatolylamine, with any of the substances capable of use in manufacturing fuchsine, but preferably with sesquichloride of carbon; this is known as diphenylamine blue. A similar blue is produced from pure phenyl paratoluidine. Commercial diphenylamine blue is probably a mixture of various substances, the chief of which are also present in Bleu de Lyons. The blue dye of Delvaux, prepared by heating rosaniline hydrochloride to 200–250° C., was probably very similar to or identical with Bleu de Lyons. Coupier's rosatoluidine blue also appears to be closely allied to Bleu de Lyons.

C. *Soluble Blue, Nicholson's Blue, Alkali Blue.*—Nicholson found, in 1862, that by treating Bleu de Lyons with sulphuric acid, and heating the mixture, a change was produced in the dyestuff, by which it was rendered readily soluble in water; the product possesses the properties of an acid, forming soluble alkaline salts possessed of much less colouring power than the free acid. Accordingly, in practice, this dye is used in a manner altogether different from those aniline dyes which consist of a compound ammonia united with some *acid*, such as hydrochloric or acetic acid; the slightly tinted *alkaline* solution of the dye (or rather of its alkaline salt) is used for dyeing, and the colour subsequently developed by passing the goods through an acid bath, when the acid itself is set free. Another soluble blue was prepared, in 1861, by Persoz de Luynes and Salvetat, by heating together 9 parts chloride of tin, and 16 of aniline, to 180° C. for thirty hours.

D. *Azodiphenyl Blue.*—Hofmann and Geyger have recently obtained a blue dye by the action of aniline on azodiphenyl diamine.

3. *Violet Dyes and Dyes Intermediate between Red and Blue.*—A. *Mauveine.*—The production of this dyestuff from aniline by Perkin, in 1856, is the era from which the manufacture of aniline colours actually dates: the oxidizing material employed by the discoverer was potassium dichromate, but it was soon found that many other oxidizing agents could give the same results. Of these the most satisfactory process appears to have been that patented in 1860 by Dale and Caro, viz., the reaction of chloride of copper (or a mixture of common salt and sulphate of copper) on an aniline salt.

Depouilly and Lauth, also Coblentz, used "bleaching" powder (Bolley, and also Beale and Kirkham, had previously found that by adding chlorine water, or bleaching powder solution, to certain aniline salts, a blue liquor was obtained, becoming purple on standing and capable of dyeing silk without mordants). According to Kopp, the product of the action of bleaching powder on aniline invariably has a redder shade than that produced by means of bichromate. G. Williams employed permanganate solution, and thereby obtained not only a purple dye, but also a more soluble red one. D. Price employed boiling dilute sulphuric acid and peroxide of lead, obtaining three different shades, according to the relative quantity of the peroxide and the aniline used: *Violine*, with one equivalent of each; *Purpurine*, with two of aniline to one of peroxide; and *Roseine*, with one of aniline to two of peroxide—two equivalents of sulphuric acid being used in each case, and twenty times as much water as aniline.

Kay obtained a product, *Harmaline*, by the action of manganese dioxide and sulphuric acid on aniline. Kopp, Stark, and also Smith prepared violet dyes by oxidizing an aniline salt with potassium ferricyanide;

Gratrix employed an aqueous solution of nitrate of copper; and Lauth, in 1860, obtained a blue violet by means of binoxide of hydrogen. Phillips used a mixture of bleaching powder and sulphate of iron. These and numerous other processes for the manufacture of violet dyes, with the exception of mauveine, have either never been adopted or have fallen into disuse.

B. *Hofmann's Violets.*—In 1863 Hofmann found that, by acting on fuchsine with methyl or ethyl iodide or bromide, dyes result of character analogous to Bleu de Lyons, but of not so blue a shade. Like the phenylated fuchsines, these dyes acquire progressively a bluer and bluer shade the further the methylation is carried, but even the final product (trimethyliodide of trimethyl fuchsine) is only a blue violet. Curiously enough, the body preceding this one in the list (dimethyliodide of trimethyl fuchsine) is a fine green; whilst all the others (methyliodide of trimethyl fuchsine, trimethyl fuchsine, dimethyl fuchsine, and methyl fuchsine) are of shades decreasing in blue cast and increasing in red shade, the nearer the body stands in the list to the original fuchsine. The dyes manufactured of this description are divided into three classes, viz., *Violet red* (*violet rouge*), marked R; *Violet blue* (*violet bleu*), marked B; and *Violet lumière*, marked B B. They chiefly consist of mixtures of methyl fuchsine and dimethyl fuchsine; dimethyl fuchsine and trimethyl fuchsine; and of nearly pure trimethyl fuchsine respectively (Girard and De Laire). Similar mixtures of the corresponding ethyl derivatives also possess nearly the same tints, but are somewhat less blue.

The term "Lumière" is technically applied to these and other analogous dyes, in order to indicate their great purity of tint; even by artificial light the colour of goods dyed therewith remains the same: ordinarily the terms "violet lumière," "bleu lumière," mean not so much special chemical compounds, as mixtures so prepared as to possess the desired quality, *i.e.*, carefully freed from any constituents which could interfere with it.

Ethylic ethers, other than the iodides, chlorides, or bromides, also give rise to violet rosaniline derivatives (probably ethylated rosanilines); thus Levinstein has prepared a dyestuff under the name "Dorothea," by the reaction of nitric ether on rosaniline.

C. *Methyl Violets* (*Violet de Paris*).—The preparation of the alcoholic iodides required for Hofmann's dyes being difficult and costly, chiefly on account of the high price of iodine, attempts were speedily made to attain the same result (viz., methylated or ethylated fuchsines) by a cheaper process. Lauth had found in 1861 that by the oxidation of methyl and ethyl aniline, dyes are obtainable of beautiful violet hues, but of not very permanent characters, the methyl aniline dyes being superior to those derived from ethyl aniline; but no practical application of this discovery was made until 1866, when Bardy succeeded in manufacturing methyl aniline, and dimethyl aniline, at a cheap rate by heating together aniline hydrochloride and methylic alcohol. The public taste in dyes having somewhat altered at this time, and colours that would stand exposure to light, &c., without alteration being no longer in exclusive demand, Poirrier and Chappat commenced the manufacture of violet and violet blue dyes, by the oxidation of the methyl aniline thus obtained.

The manufacture of these violets is now carried on to a large extent, and there seems reason for supposing that the trade will continue. There are certain differences noticeable between the two classes of dyes that in various cases lead to the preference of the one rather than the other, according to the taste or practical experience of the dyer. Recent experiments of Hofmann ("Deut. Chem. Ges. Berichte, vi. 352) tend, however, to show that the products are chemically identical.

D. *Miscellaneous Violets and Mauves.*—These are for the most part formed by processes substantially the same as those by which these two main dyes are produced, the difference being in the nature of the body chosen to modify the rosaniline. Thus:—Wanklyn suggested the use of isopropyl iodide; Perkin of nascent methyl and ethyl bromides produced by the action of heat on turpentine and bromide in presence of alcohol, and Lauth and Grimaux the use of benzyl chloride. Wise obtained a violet by acting on fuchsine with valeric acid; Kopp heated tannate of fuchsine with wood spirit containing hydrochloric acid, thus probably obtaining Hofmann's methyl violet by the action of methyl chloride. Smith heated fuchsine with salicylic acid, obtaining a fine violet; he also employed brominated acetone derivatives, forming an analogous colour.

The Regina purple or Nicholson purple, patented by Nicholson in 1862, was prepared by heating rosaniline salts to about 210° C., and has probably an analogous composition. Delvaux obtained a somewhat similar substance, together with a red dyestuff, by heating aniline hydrochloride with sand to 200°–220° C.; the red product was soluble in water; the violet body being extracted from the residue by means of alcohol.

In 1867 Girard, De Laire, and Chapoteau, published the results of an investigation on the bye products of fuchsine-making; from these they extracted a violet and a mauve dye, termed by them respectively *violaniline* and *mauvaniline*. The same bodies give rise to various blue violets by operating on them with methyl iodide, &c., in the way that Hofmann's violets are made. Previously to the publication of these researches Schneider had found that a violet dyestuff is produced along with the "azaleine" prepared by means of mercuric nitrate; and Persoz, De Luynes, and Salvetat had found a similar substance in the product of the action of tin tetrachloride on aniline; Paraf had also published a process for obtaining from fuchsine residues red, puce, and violet dyestuffs (Bull. Soc. Chem. Paris, [2] vii. 92.)

In 1865 a violet was sold under the name of Dahlia Imperial,* said to be obtained from fuchsine residues; it gave shades of great beauty, and commanded a high price. A similar dyestuff is obtained by the action of iodide of ethyl on mauveine.

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* The name Dahlia has also been applied to various other products.

STÆDELER, in 1866, found that perfectly pure aniline gives, on treatment with arsenic acid, a violet substance soluble in alcohol (Bull. Soc. Chim. Paris, [2] v. 225). A violet dye is also obtainable by the action of heat on a mixture of aniline hydrochloride and nitrobenzene, or by heating together at 180°–195° C. for from four to six hours, the following mixture:—Aniline (from benzol boiling constantly at 81·5° C.), 10 parts; nitric acid at 40° B, 3 parts; hydrochloric acid, 12 parts. Probably these products are all essentially violaniline. (COUPIER, *ibid.* vi. 500.)

By the action of nitrous acid on aniline, a product (azodiphenyl diamine) results, which, when phenylated, forms a blue or violet dyestuff, isomeric or identical with violaniline (HOFMANN and GEYGER). This reaction has not yet come into use as a trade process; the term "azodiphenyl" blue has, however, been applied to the product.

4. *Green Dyes.*—A. *Miscellaneous.*—It has been long known that certain aniline salts, especially the nitrate, turn green on the outside by exposure to air, probably by oxidation. WILLM found that when potassium chlorate is allowed to act on violet (mauveine?) made by DEPOUILLY and LAUTH's process, a green precipitate is formed, and a liquid which communicates a green tint to wool dipped in it, and afterwards exposed to the air. BEALE and KIRKHAM also found that by the continued action of chlorine or of bleaching powder on an acid solution of aniline, a green was formed, probably by further oxidation of the violet first produced. In 1860 CALVERT, LOWE, and CLIFT attempted to utilize this reaction for colour printing, and termed the tint produced by the action of potassium chlorate on aniline salts, *Emeraldine:* this name has also been applied to aldehyde green.

Green colours were also made by mixing picric acid, or other yellow dyes, with aniline blues or purples; these mixtures, however, were unsatisfactory in tint by artificial light, looking dull and greyish. The picrates of triethyl rosaniline and trimethyl rosaniline have likewise been thus used to a considerable extent.

B. *Aldehyde Green* was first made in attempting to fix aldehyde blue: for this purpose CHERPIN made use of thiosulphate (hyposulphite) of sodium (it is said by the advice of a photographer who considered this agent the universal fixer). LUCIUS subsequently employed sulphuretted hydrogen solution, and then a solution of sulphurous acid to develop the green tint; USÈBE, sulphide (not polysulphide) of sodium; and HIRZEL sulphide of ammonium. SCHLUMBERGER prepared a similar dye by treating rosatoluidine, dissolved in sulphuric acid, with aldehyde.

C. *Iodine Green.*—During the manufacture of HOFMANN's violets a green colour is sometimes produced along with the violet products; but until about 1865 this substance was not specially prepared or isolated. In that year WANKLYN patented a process for its production; FILLMANNS, and also MEISTER LUCIUS and BRÜNING, had, however, prepared it for the market previously to this. At first it was sold as a picric acid or tannin compound, nearly insoluble in water, and for dyeing purposes was dissolved in alcohol. Of late years the "iodine green" of commerce has been divided into several kinds; thus, instead of the picric acid compound, a zinc chloride compound has been prepared which is soluble in water. Frequently the dyestuff has been sold as liquid or as a paste readily soluble. In order to save the iodine the iodized body has also been converted into the corresponding chlorinated derivative, which dyes equally well.

D. *Methyl Green.*—This body is in all probability identical with "iodine green;" in any case it only differs from it in the same way that Violet de Paris differs from HOFMANN's methyl violet. At first it was produced from Violet de Paris just as iodine green itself was, viz., by heating with the iodide of methyl (ethyl isopropyl, amyl, &c., &c.), the green dye being essentially a dimethyl iodide of a trimethylated fuchsine; but latterly the use of methyl chloride has superseded that of methyl iodide. The green is sent out in crystals, solution, or a paste consisting either of dimethyl chloride of trimethyl fuchsine, or of the double chloride of zinc and this body, or of some such compound.

Perkin's Green is stated to be a magenta derivative, more closely resembling iodine green than aldehyde green, but differing from it in being more soluble, and also in being precipitable by means of carbonate of sodium: it is chiefly used for calico printing.

5. *Miscellaneous Dyes.* A. *Aniline Black.*—The various aniline blacks in use are not strictly speaking dyes; they are almost invariably produced during the process of colour printing, or by some subsequent treatment of the printed goods, and are not, like the true aniline dyes, in a state fit for immediate use by the dyer. Aniline black is in fact, at least in many instances an intensely dark green.

In 1862 LIGHTFOOT printed fabrics with a mixture containing *inter alia* chlorate of potassium, an aniline salt, and copper chloride; by the reaction of these salts on each other, facilitated by exposure to air, black is developed. The copper salt, however, acted injuriously on the steam-printing rollers, and the mixture could not long be kept. Subsequent researches by LAUTH and others showed that though copper compounds are essential to the production of the colour, they need not be soluble; by substituting sulphide of copper for chloride the injurious action on the rollers is for the most part avoided. VERSMANN states that in order to prepare a good black, aniline as free as possible from higher homologues should be used, the purity of the shade being exactly proportionate to that of the aniline. The number of modifications of the processes of LIGHTFOOT and LAUTH is almost innumerable; each printer has his own pet formula. The general belief is that no good black can be formed without copper. KOECHLIN, however, states that the oxidation of a solution of aniline in hydrochloric acid by a mixture of potassium chlorate and ferricyanide yields a good black dye. He also recommends the use of tartrate of aniline mixed with sal ammoniac, in lieu of a mineral salt, such as the hydrochloride of aniline—injury to fabrics, mordants, &c., being thereby avoided. By the further action of bichromate of potassium on goods printed with aniline black, the purity of the black tint is much improved, and more solidity given. Blumer Zweifel states, that if an iron salt is substituted for a copper salt in the aniline black mixtures, a dark *indigo blue* is produced, and not a black at all. Bolley recommends this process for printing calicoes with indigo blue tints.

B. *Yellow and Orange Dyes.*—The chief of this class are the chrysaniline of Hofmann and the chrysotoluidine of Girard, De Laire, and Chapoteau; these are commercially known as "Phosphine," "Victoria orange," "Aniline orange," "Aniline yellow," &c., and are probably identical in the main, the orange shades being communicated by a slight admixture of fuchsine. They are extracted from the bye products of the fuchsine manufacture. Besides these are various dyestuffs of minor importance, such as *Zinaline*, obtained by Vogel by the action of nitrous acid on fuchsine, various yellows from the action of nitric acid on rosaniline and its homologues, and various yellows and oranges from the action of nitrous acid on aniline. Scheurer-Kestner prepared a yellow dyestuff by the action of tin and hydrochloric acid on mauveine. Oxidizing agents transform this body into a red product, and goods dyed with it become first orange and then red by exposure to air.

C. *Aniline Browns. Phenylene diamine Brown.*—By reducing dinitrobenzene, phenylene diamine results, and by acting on this with a nitrite a brown colouring matter results. It dyes silks and wools without mordants.

Wise's Brown.—Fuchsine and formic acid when heated together for some hours give an orange red colouring matter, which when heated with aniline becomes chestnut.

In addition to these, brown dyestuffs are obtained from leucaniline and chrysaniline; Girard & De Laire, Sieberg, Jacobsen, and others, have prepared brown dyestuffs by processes, descriptions of which are given further on.

D. *Aniline Greys.*—Castielaz has prepared a grey dyestuff by acting on mauveine with sulphuric acid and aldehyde; other greys have also been described by Block, and also Carver and Therault.

MANUFACTURE OF ANILINE DYES.

The limits of the present article only permit a description of those processes which have been successfully worked on the large scale; and although chronologically the dye known as mauve or Perkin's violet comes first in order, it is more convenient to discuss the dyes according to their colours, beginning with fuchsine as the most important, firstly, on account of the large consumption of that dyestuff as such, and secondly, on account of its importance as the raw material in the manufacture of many other dyes.

Manufacture of Fuchsine.—Verguin's process was worked by MM. Rénard Frères and Franc, as follows, in accordance with their patent of April 8, 1859:—Into an enamelled iron pot holding some 20 kilos. were introduced 10 kilos. of aniline oil, and then 7 of tin tetrachloride, in small portions at a time, with constant stirring; the whole was then heated under a hood on a small fire until it boiled gently; the mass gradually darkened in colour, becoming deep red, and finally (in about half an hour) appearing almost black. When the required depth of shade was attained, the fluid was poured out upon cooling plates, where it solidified to a mass containing fuchsine corresponding to about 5 or 6 per cent. of rosaniline hydrochloride, aniline hydrochloride, salts of tin, and bye-products. Although the yield was small, the tint of this product was good; no purification was attempted, the crude melt being sent direct into the market under the name of fuchsine.

The azaleine of Gerber-Keller was prepared by heating together 10 parts of aniline, with 7 to 8 of finely powdered dry mercuric nitrate; the liquid became gradually red, the operation being interrupted when the mass began to thicken and emit yellow vapours. The whole was then poured into water, when the unaltered aniline floated to the top; the undissolved portion was again treated with water, and from the solutions thus obtained the dyestuff was thrown down by addition of common salt. A much larger yield was obtained than by Verguin's method; the mercury was almost wholly regained in the metallic state, whilst the nitric acid could also be recovered from the liquors. To avoid deflagration, and to obtain the best yield, Gerber-Keller recommends the operation being conducted on the water bath (*au bain-marie*); mercuric nitrate being added in small portions from time to time with constant stirring for 8 to 9 hours.

Another method is to heat a mixture of aniline and a highly concentrated solution of mercuric nitrate, free from excess of nitric acid, to 120° C. for from 10 to 18 hours. Metallic mercury sinks to the bottom; the resulting fluid is transferred to an iron still, and unaltered aniline distilled off by blowing steam through the liquor; the dyestuff is precipitated from the remaining solution by common salt, and then recrystallized.

The dyestuff made by means of mercuric nitrate is still in the market, though not in such large quantities as that made by means of arsenic. It is sometimes designated as *Rubine* or *Rubine imperial*, and being free from arsenic, is sold at a high price. Confectionery, sausages, wines, and liquors, are often tinted with it. The ordinary arsenical dye is sometimes used for these purposes, and traces of arsenic can often be detected in the substance thus coloured. The essential difference between azaleine or rubine and the fuchsine of Verguin, is that the latter is a hydrochloride, the former being a nitrate of the same base.

In an English patent taken out by Perkin, the mixture of aniline and mercuric sulphate or nitrate used is recommended to be heated to about 175° C.

Lauth and Depouilly's method depends on the action of heat (150°–160° C.) on a mixture of aniline nitrate, 1 part, and free aniline, from 6 to 8 parts; but it is somewhat difficult to avoid rise of temperature and consequent deflagration. The yield by this process is from 7 to 8 per cent. as a maximum, and the product is much more impure than that obtained by the preceding methods. From the crude melt a purified dyestuff is obtained, by boiling with twenty times its weight of water; unaltered aniline carrying fuchsine nitrate with it rises to the surface; this is skimmed off and filtered whilst hot through sand, and crude fuchsine nitrate is obtained on cooling as a greenish pasty mass with metallic lustre. Either benzol (boiling between 60° and 100° C.) or ether may be used to dissolve out the aniline, chrysaniline, mauvaniline, &c. The residue is cooled with caustic soda or potash ley, the aniline nitrate is decomposed, aniline being carried off by the steam. The mixture of bases (rosaniline, violaniline, &c.) is then treated with dilute hydrochloric acid, which takes up chiefly the rosaniline; on cooling the liquors deposit rosaniline hydrochloride.

Notwithstanding the considerable difficulties in the way of making this process work commercially, it has been from time to time revived and repatented with but slight modifications; thus Hughes patented a method the same in principle—viz., heating together a mixture of aniline oil, 20 parts, and nitric acid of specific gravity 1·36, 6½ parts. After the water has distilled off, the materials are kept at 150°–200° C. for an hour, gently boiling; a dark red syrup is thus obtained, which is treated with water and soda to neutralize free acid, and then well washed to remove unaltered aniline nitrate.

In 1862 Laurent and Casthelaz prepared *erythrobenzol* by mixing together 12 parts of nitrobenzol, 24 of iron filings, and 6 of ordinary hydrochloric acid, and allowing the mass to stand for 24 hours. The production of colour is much facilitated by heating the mixture. Bolley was unable to obtain any colouring matter by action in the cold. Coupier has proposed a very similar method, consisting in the heating of a mixture of aniline ferrous chloride and nitrobenzol to 200° (see *infra*).

The carbon tetrachloride process was thus worked by Monnet and Dury:—A digester furnished with a safety valve and a thermometer was heated, first to 116°–118° C., by superheated steam, and finally to 170°–180° C. After cooling the mass was extracted whilst still warm, and boiled with water so as to get the dyestuff in solution. The proportions used were aniline oil, 4 parts, and carbon tetrachloride, 1 part. The yield is stated to be much superior to that obtained by means of tin chloride.

Delvaux heated a mixture of dry aniline hydrochloride 100 parts, aniline oil 72 parts, and dry sand 1000 parts, to 110°–120° C. for 15 hours. On boiling the product with water, a large quantity of fuchsine is dissolved out as hydrochloride; the remainder can be extracted by alcohol, or by first boiling with an alkali and then with dilute hydrochloric acid. Lauth has proposed a similar process for the preparation of Paris violet from methyl aniline.

Smith's antimony process is thus carried out: 6 parts of antimony are heated with 29 of pure nitric acid of sp. gr. 1·44, added in small portions, from half to three quarters of an hour being required for 3 kilos. of antimony. The vessels used are of earthenware, fixed inside cast-iron pans with Roman cement. The nitrous fumes given off are utilized for vitriol making. Constant stirring throughout the oxidation process is necessary; finally a white, very acid, and slightly damp powder is obtained. This is heated for 12 hours till dry, and is then calcined at a dull red heat in cast-iron vessels, in batches of 50 kilos., till all free nitric acid and moisture is driven off. A yellow powder of antimonic anhydride then results.

Eight vols. of aniline are mixed with nine of commercial hydrochloric acid of sp. gr. 1·165, and the whole evaporated on a sand bath till thick white vapours are evolved; it is very essential that no free acid should be here present. This can be readily effected by an exact neutralization of the acid by aniline; the point of neutrality being determined, not by the ordinary test papers, but by means of paper soaked in fuchsine solution. A violet or bluish tint is given to the paper by a trace of acid.

Twenty-five kilos. of dry aniline hydrochloride are charged into an earthenware conical pot, set in an iron pan heated to 240° C. by a paraffin bath. When thoroughly melted, 32 kilos. of antimonic anhydride are added in four batches, hour by hour with constant stirring. A tolerably energetic action is set up, but this soon subsides, and altogether ceases in from five to six hours. The "melt" is then ladled out by copper ladles and allowed to cool.

During the melting from 2½ to 3 kilos. of free aniline, and 2 to 2½ kilos. of water pass off; these are condensed by a head connected with a condensing worm, a draught being kept up by placing the free end of the worm in connection with a chimney.

The "melt" is powdered and mixed with 22½ kilos. of coarsely powdered soda crystals, and 30 litres of water; carbonic acid is disengaged. The whole is then heated to 80° C. for an hour with continual stirring, allowed to settle, the deposit strained on calico, and slightly washed with cold water. The moist solid mass is then heated with 300 litres of water in a copper boiler by means of steam. The solution, after settling, is filtered into crystallizing pans. The residue is again treated with 200 litres of water, and the liquor also filtered into crystallizing pans. The insoluble portion is boiled with another 100 litres of water, and the colour precipitated from the solution by common salt; and finally the residues are exhausted with water, and the liquors used, together with the mother liquors from the crystallizing pans, for the treatment of a fresh batch. When the impurities become so concentrated in the liquor that the dye will not crystallize properly, but requires to be precipitated by common salt, the antimony is regained from the insoluble residue by heating with carbonate of soda, common salt, and charcoal, in a specially constructed furnace. Two-thirds of the antimony used is thus regained in the metallic state. The yield of moist fuchsine obtained is about 7 to 7½ kilos., in certain cases as high as 12 to 12½ kilos., containing about half its weight of dry colour. Some varieties of aniline oil require more antimonic anhydride than others; thus, for some kinds equal weights suffice, whilst for others 25 parts of aniline hydrochloride require 35 of anhydride. (Sieberg, *Dingler's Pol. Journal*, clxxi. 366.) Reimann states that this process is very long and inconvenient, and is, in fact, "of no value," as useful magenta cannot be made by its means; the manufacture of magenta in this way was, however, carried on by the inventor, in Glasgow, on a large scale for several years (several cwts. of aniline being worked up daily).

The reaction of nitrobenzol on aniline, or an aniline salt, appears to be commercially applicable as a means of producing fuchsine, though till recently it has not found much favour; certain metallic salts greatly facilitate the reaction. Holliday heated to 227° C. for several hours a mixture of aniline of high boiling point 2 parts, commercial hydrochloric acid 2 parts, and nitrobenzol about 1 part, and obtained a "melt" containing much fuchsine.

Staedeler has published the results of several experiments on the reaction of nitrobenzene on aniline and toluidine, showing that blue, violet, and rose colouring matters are readily obtainable, according to the nature and proportions of the materials used and the heat employed in its duration.

Coupier has patented, amongst other processes, the heating together of the following mixtures for the production of rosatoluidine, rosaxylidine, and violaniline products respectively:—(1.) "Coupier's nitrotoluol" (a mixture of para- and meta- nitrotoluene), 95 parts; Coupier's toluidine (a mixture of para- and meta- toluidine), 67 parts; hydrochloric acid, 65 parts; and ferrous chloride, or an equivalent quantity of ferric chloride, 7 parts. (2.) Coupier's xylidine, 75 parts; and Coupier's nitroxylol, 105 parts.

Schutzenberger states that the dyestuff obtained by process (1) is identical with ordinary rosaniline, whilst the yield is as great, if not greater (*Jahresbericht*, 1869, 1162). Greiff, on the other hand (*Ibid.* 1163), found that much resinous matter was formed, and also a large quantity of a dirty violet product.

Lauth adds stannous chloride to a mixture of aniline and nitrobenzol, when an energetic action ensues. By adding boiling water when the tint becomes dark brown, a solution containing much fuchsine is obtained.

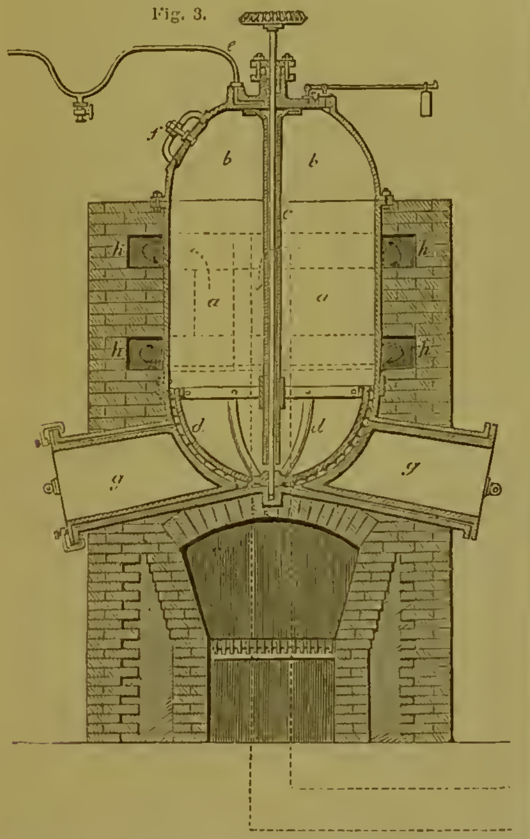
Meister Lucius and Brüning have recently revived the manufacture of fuchsine by the reaction of nitrobenzol on aniline, and are now sending into the market considerable quantities of fuchsine made in this way. Like the rubine prepared by means of mercuric nitrate, this dyestuff is *free from arsenic*, and hence is specially suitable for the artificial coloration of edible substances. Their process of manufacture is stated to differ from that of Coupier in certain particulars, although based on the same principles.

Medlock's process for the manufacture of fuchsine is by heating 2 parts of aniline with 1 of "dry arsenic acid." His specification was so worded that his process, as actually patented, was practically valueless, and considerable litigation was the result. Arsenic anhydride, As_2O_5, appears to be, in fact, incapable of transforming aniline oil into fuchsine; in order to form this dyestuff it is essential that a hydrated oxide of arsenic should be present *i.e.*, that the elements of water should be present, associated with those of arsenic anhydride, forming what is now spoken of as arsenic acid, *e.g.*, $As_2O_5,3H_2O$, or H_3AsO_4. A very concentrated aqueous solution of this substance is used, the excess of water being volatilized before the reaction is completed.

The arsenic acid was formerly prepared by acting on white arsenic (As_2O_3) with nitric acid. A cheaper process is to treat the arsenious oxide suspended in water, or preferably dissolved in hydrochloric acid, with chlorine; it is usually employed in the form of a syrupy liquid, containing from 70 to 75 per cent. of As_2O_5. The first method adopted was to heat a mixture of about 100 parts of aniline oil, and 150 to 160 of this acid (or a proportionally larger quantity of a weaker acid) in an iron pot, preferably enamelled internally, set either in a sand or hot-air bath, or better still, in an oil bath, and covered with a hood connected with a flue, to avoid injury to the workmen by the vapours evolved. The heat being gently raised with continual stirring, water was evolved, together with some aniline vapours; the mass gradually acquired a deep brownish red tint, becoming thicker and thicker, the temperature gradually rising to a point between 150° and 190°, too high a temperature being avoided as prejudicial to the yield and purity of the colouring matter. From time to time samples were drawn, and when these presented the right appearance, possessing after cooling a metallic bronzed tint, and breaking with a clean shining fracture, the heat was discontinued; the product (technically called the "melt") being ladled out on to iron plates to cool. When solid, the cakes were pulverized and then subjected to further treatment with solvents to extract the red colouring matter.

Numerous improvements and modifications have been made in this first rough process. In the first place, the aniline volatilized was a considerable source of loss; this was avoided by applying a cover and condensing worm to the pot. The next improvement was to use larger vessels, in which, by applying mechanical agitators moved by steam power, a much more uniform quality of product could be obtained. The ladling out of the "melt" while yet fluid, and the pulverization of the solidified mass, constantly exposed the workmen to the injurious action of the vapours; again, the presence of unaltered aniline in the "melt" is prejudicial to the effectual extraction of fuchsine, or, at least, lengthens the process, the final products (rosaniline salts) being less easily purified by crystallization in presence of aniline salts than in their absence. Besides these imperfections, it frequently happened that unsuitable aniline oils were employed, occasioning a great deficiency in the yield. Thus Nicholson and other manufacturers soon found practically that a body containing nearly 100 per cent. of pure aniline (*i.e.*, an oil made from a benzol boiling at 81° or thereabouts, and boiling at close upon 182° C.), was not the best material to operate on; an oil distilling for the most part between 185° and 200° C. being more suitable. Until Hofmann's researches had cleared up the composition of fuchsine, the *raison d'être* for this was unknown, although the fact was certain.

Fig. 3.



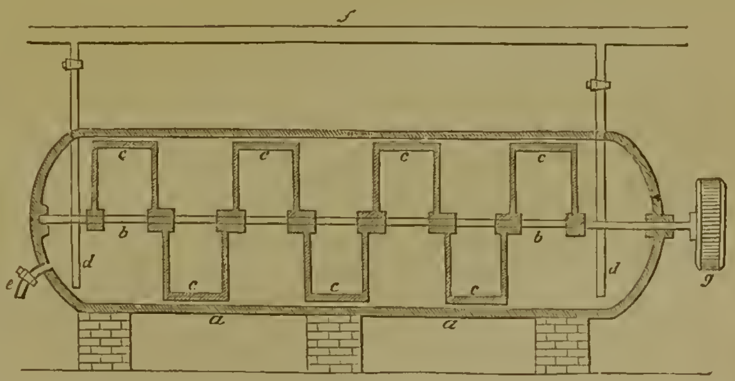
The improved method employed at the present day is thus described by Girard and De Laire. Into a retort holding about 2500 litres are introduced successively 800 kilos. of aniline oil (either prepared by partial fractional distillation of benzol, so as to produce directly a suitable aniline, or by mixture of various kinds of aniline oil, so as to obtain an oil of suitable composition), and 1366 kilos. of acid, containing 72 per cent. arsenic anhydride (this solution will not solidify even in the winter). An agitator moved by steam is provided, the axle of which is hollow, so that steam can be admitted to the bottom of the retort through the spindle of the agitator itself. In the base of the retort are two doors for withdrawing the finished charge, and at the top a safety valve, and a tap connected by a pipe with a tank of hot water. Fig. 3 indicates the general disposition of the apparatus:—*a*, *a*, the body of the retort; *b*, *b*, the dome; *c*, the hollow axle of the agitator; *d*, *d*, the serrated arms of agitator, to prevent the material from sticking to the bottom of the retort; *e*, the steam tube; *f*, the manhole; *g*, *g*, openings for withdrawing the charge; *h*, *h*, hot-air flues for heating the retort: these can be opened so as to cool the retort in case of necessity. The neck communicating with the condensing worm is on the other side of the apparatus, and is not shown in the section.

The retort is heated by hot-air flues, and is provided with a good condensing worm to condense the aniline and water that pass over; the temperature should not rise above 190°–200° C. In from eight to ten hours the operation is completed, the progress being judged by measuring the bulk of the condensed liquor. This should amount to about 850 litres, and after saturation with salt about 440 kilos. of aniline and 410 litres of water should be obtained. When 800 litres of distillate have passed, and the operation is nearly finished, the fire is withdrawn, the residual heat of the furnace being sufficient to finish the distillation of the unchanged aniline. To separate the last traces steam is blown through the "melt" in the still, which mechanically carries over the last portion of the aniline vapour. A small quantity of hot water is then run in, and then more in small quantities at a time; heat may be reapplied to the retort to facilitate the "hydration" process. At the end of about an hour a homogeneous fluid mass is obtained, which is run off through the openings into the solution-vats for the next operation.

By this process great economy in labour, fuel, and cost is effected; and in addition, the operation of pulverizing the cake of solidified melt is avoided, so that the workmen are in no way exposed to the chance of inhaling arsenical dust, or to the other painful consequences produced by the local action of solid arsenical matters (sores, ulcerations, &c.) Four men can thus work off 2000 kilos. of crude material per diem, and of these three are only required at the beginning and end of the operation; by the older processes ten men are required to turn out as much material.

In many factories, instead of admitting steam at the end of the operation to distil off the last traces of aniline, and running in hot water to avoid the production of a solid cake, the "melt" is run out on to cooling plates, placed under a hood to carry off the aniline vapour. The cake is then transferred to a boiler provided with an agitator, Fig. 4. *a*, *a*, is the

Fig. 4.



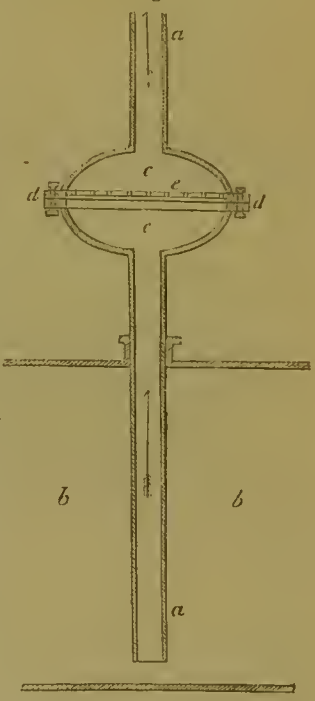
boiler set horizontally; *b*, *b*, is the spindle, and *c*, *c*, are the vanes of the agitator; *d*, *d*, the steam pipes for heating contents of boiler; *e*, the discharging cock; *f*, steam tube; and *g*, the cogwheel communicating motion to the agitator. When worked up with hot water (or other solvent) the dyestuff is extracted without necessitating a previous pulverization of the cake. Lixiviation, after the fashion of the alkali manufacture, is ineffective for fuchsine-making, the insoluble matter being of a viscid resinous nature. Great loss of dyestuff is occasioned by the mechanical covering up of its particles, unless a continuous agitation is kept up; even as it is, the insoluble residues left after treating the crude fuchsine often contain 3 to 4 per cent. of rosaniline.

As before stated, in the first days of fuchsine-making the crude melt was often sold to the dyer, who simply dissolved it in hot water, filtered, and used the liquor. A demand for a purified article, however, soon sprung up. At first the colour maker contented himself with simply extracting the soluble dyestuff with boiling water, filtering through beds of sand, and precipitating the clarified liquor with common salt in excess. A mixture of arseniate and hydrochloride insoluble in brine was thus thrown down. It was soon found that hydrochloric acid was a much better solvent than plain water. The liquors obtained by this means were precipitated by common salt, or by addition of just sufficient soda to saturate the acid and form brine, and the precipitate, consisting mainly of fuchsine hydrochloride, was redissolved and recrystallized; or they were precipitated by lime, soda, &c., in excess, and the precipitate dissolved up in some other acid (*e.g.*, nitric, oxalic, or acetic), and the corresponding salt thus formed was purified by crystallization. The arsenious chloride vapour evolved by the hydrochloric acid, and the aniline vapours thrown off by the action of the alkalies, were highly injurious to health.

Habedank proposed the following modification of the process for obtaining the hydrochloride (*Dingler's Pol. Journal*, clxxi. 73)—1 part of "melt" and 5 of water are used, and as much common salt as is equivalent to the arsenic acid originally employed; these are thoroughly boiled together, when rosaniline hydrochloride is formed, insoluble in the saline liquor containing sodium arsenite and arseniate. This liquor is run off and the resinous mass thoroughly boiled with water: an impure solution of rosaniline hydrochloride is obtained, filtered, and then allowed to crystallize, and the crystals purified by repeated crystallization. By this process the rosaniline arseniate and arsenite are converted into hydrochloride without the evolution of arsenical vapours.

The filtration of the liquors is best effected by means of the following device (Fig. 5)—ordinary filters are practically useless, as they rapidly become clogged by the viscid insoluble matter. A tube, *a*, *a*, dips down nearly to the bottom of the boiler, *b*, *b*; on this tube is a nearly spherical expansion, *c*, *c*, consisting of two concave iron vessels capable of being united together by a flange and screw bolts, *d*, *d*: between the mouths of these two vessels is strained a sheet of felt or other filtering material, a perforated iron plate, *e*, being fixed above to prevent rupture of the filter cloth. The boiler is so constructed that it can be made steam tight; when steam is admitted the pressure forces the liquid up the pipe, *a*, *a*, and through the filtering cloth upwards to a receiving tank. The tarry matters being thus left on the *under* side of the filter drop off by their own weight and fall back into the boiler. A set of these filters is so arranged that any one can be shut off, the hemispheres opened, and a new filter-cloth substituted for the old one without interfering with the action of the others. Differently shaped filters constructed on the same principles are frequently employed.

Fig. 5.



The liquid mass obtained by the improved process, or the crude "melt," is usually treated in one of the following ways, viz.: — (1) Boiling with hydrochloric acid, and precipitation of the dyestuff with sodium carbonate, as described below (chalk, limestone, or marble powder, may also be used instead of carbonate of soda). (2) Boiling with water alone, or preferably with water very feebly acidified with hydrochloric acid, and subsequent filtration and treatment of the filtrate with common salt. (3) Boiling with water and excess of lime, chalk, or soda. In each of these processes the boiling is best effected under pressure, time and labour being saved, and more concentrated solutions obtained.

For the first method the following proportions may be taken:—Solid "melt," or its equivalent of liquefied mass, 11 parts; hydrochloric acid, 2 parts; water, 33 parts. Steam is passed through for three or four hours, or until the residue becomes pulverulent and contains little or no red soluble matter; the liquor is then filtered and treated with sodium carbonate in quantity just equivalent to the sum of the arsenic and hydrochloric acids present. Rosaniline hydrochloride is precipitated, being insoluble in the resulting soda liquors, and floats to the top, being buoyed up by the carbon dioxide bubbles formed, and is skimmed off, dissolved in water by means of steam, and crystallized. The first crystals are redissolved in a boiler provided with an agitator, and the solution filtered and allowed to crystallize. The residue left on the filter consists for the most part of humus-like bye products along with mauvaniline, chrysotoluidine, and sometimes lakes formed by the presence of alumina in the sodium carbonate used. Violaniline is not present, its hydrochloride being practically insoluble.

. The mother liquors arising from the recrystallization are often used over again in place of water, either to redissolve the first batch of crystals, or to treat the crude "melt." In this way the chrysotoluidine taken up together with the rosaniline is concentrated in the liquor (chrysotoluidine salts are more soluble than rosaniline salts in slightly acid solutions); and as one result the fuchsine made is apt to contain a little of this substance, giving it a scarlet shade. In some factories the mother liquors are precipitated by common salt, and the scarlet or crimson dyestuff thus thrown down (consisting of fuchsine more or less mixed with chrysotoluidine) is sold under the name of *Cerise*, *Scarlet Magenta*, &c., or is kept apart for the manufacture of certain dyestuffs made from impure fuchsine, *e.g.*, browns, greys, &c., &c.

For the second method, the following proportions may be used:—Crude fuchsine, 100 parts; hydrochloric acid, 1 part. Steam is blown through for four or five hours, and the resulting liquors filtered into large tanks: 120 parts of common salt is then added in small quantities, the liquid being agitated; the rosaniline hydrochloride thus thrown down is slightly washed and recrystallized as before.

For the third method, the one most generally adopted at the present day, the crude fuchsine is thoroughly boiled with an excess of lime, and a sufficient bulk of water to dissolve all the rosaniline present, for several hours: in case a "melt" has been used not previously freed from undecomposed aniline salts, the aniline is by this means liberated and expelled. Solid arseniate and arsenite of calcium are formed and separated by filtration while boiling hot; on cooling, a crop of crystals of impure rosaniline in the free state is obtained. The mother liquors may be used for the treatment of a fresh batch, or may be employed for the preparation of inferior qualities of dyestuff. It usually happens that a certain quantity of rosaniline is left in the lime mud; this is most completely extracted by treatment with dilute hydrochloric or sulphuric acid, and precipitation of the rosaniline solution by common salt. The first crystals of rosaniline may be purified by another crystallization from water; or they may be treated with twice their weight of alcohol, and the filtered solution distilled (mauvaniline is thus left undissolved, whilst the chrysotoluidine is only partially dissolved). Usually, however, these crystals are converted into some rosaniline salt, *e.g.*, the hydrochloride, and this salt purified by crystallization. A slight excess of acid being used, the chrysotoluidine is left in the mother liquors, whence it is sometimes extracted for sale under the names of Chrysaniline, Aniline yellow, Victoria orange, &c. Some manufacturers employ chalk, baryta, or soda in place of lime.

Yield of Rosaniline.—The amount of crystallized salt obtainable from a given quantity of aniline oil necessarily varies with the nature of the oil, and the way in which the manufacture is effected. By the improved process for fuchsine-making, coupled with the hydrochloric acid and carbonate of soda process for dissolving out and purifying the rosaniline hydrochloride, for every 100 parts of aniline originally employed from 25 to 30 parts of dry crystallized rosaniline hydrochloride can be obtained (GIRARD and DE LAIRE). According to REIMANN, 34 lbs. of magenta (rosaniline hydrochloride?) may be obtained from 1 to 2 cwts. of aniline oil of suitable composition. KLETZINSKY has, however, stated that from 100 parts of aniline and 200 of arsenic acid, 50 parts of pure crystallized red are obtainable (*Jahresbericht*, 1864, 820).

By the process of R. SMITH with antimonic anhydride, 100 parts of aniline oil yield from 30 to 50 of moist paste of colouring matter, containing half its weight of dry hydrochloride, from 10 to 12 parts of aniline being regained during the heating process by condensation of the vapours evolved (SIEBURG). This, therefore, represents a yield of between 17 and 29 parts of hydrochloride per 100 of aniline actually used. BOLLEY states that 40 per cent. of the weight of aniline used may be regarded as a very high yield of crystallized fuchsine, and 33 per cent. as an average amount. ROSENSTIEHL states that the yield of dry crystallized aniline red obtainable on the large scale averages from 27 to 30 parts per 100 of aniline used, whilst LEVINSTEIN estimated it at 20 parts; probably 28 per cent. may be taken as a fair average yield. The mercuric nitrate process is stated to yield, when properly conducted, quite as large an amount of crystallized fuchsine as is obtainable by means of arsenic acid under the most favourable circumstances. According to MONNET and DRURY, the yield of red dyestuff by the carbon tetrachloride process will compare favourably with that obtainable by any other method.

SCHUTZENBERGER states that by the reaction of aniline oil on commercial nitrobenzol a yield is obtained quite as great as that by means of arsenic acid, if not greater; COUPIER says that by this process a yield as high as 40 per cent. of crystallized colouring matter can be obtained, and that this dyes shades twice as dark as those given by an equal weight of ordinary commercial fuchsine.

Preparation of Rosaniline Salts.—As mentioned above, Azaleine and Rubine are virtually the nitrates of rosaniline. From the hydrochloride of rosaniline the free base is commercially prepared by dissolving the salt in a large bulk of boiling water, adding an excess of boiling alkaline solution, and continuing the boiling for some hours; the liquor is then filtered while hot into crystallizing vats, where the free base separates on cooling as nearly colourless crystals. Care must be taken that there is sufficient water present to retain the whole, or almost the whole, of the free rosaniline in solution; to avoid inconveniently large bulks of liquors, the solution is best effected under pressure.

To form the acetate, rosaniline is dissolved in boiling acetic acid, containing one-fifth as much glacial acid as there is used rosaniline, two and a half times as much water being also present. To obtain an easily crystallizable salt, glacial acid, free from sulphurous or sulphuric acid, should be employed; the rosaniline should be itself well crystallized, or at any rate prepared from a salt several times crystallized, and perfectly dry and in fine powder: it must be free from every trace of excess of fixed alkali used as precipitant. 100 parts of rosaniline are placed in an enamelled iron pan, and 20 of the glacial acid added little by little with continual stirring; the mass may be heated to 60° or 70° C. with advantage, but care must be taken that the acid does not distil over. When thoroughly incorporated, 240–250 parts of boiling water, free from calcareous salts, is rapidly added, the whole boiled a few minutes (but not long, otherwise the acetate is partially decomposed, an uncrystallizable basic salt being formed), and then run into crystallizing pans. After two or three days fine crystals are obtained, the weight of which is about equal to that of the rosaniline used.

It is possible to prepare the acetate by double decomposition of the sulphate, arseniate, &c., by lead acetate; but the resulting salt is less finely crystallized. The insoluble lead compound retains a considerable amount of colouring matter, and the yield is greatly diminished.

The sulphate of rosaniline may be prepared in the same way as the hydrochloride, substituting, however, sulphuric for hydrochloric acid if method No. 1 be used, or sulphate of soda for common salt if method No. 2 be employed; or it may be prepared by adding sulphuric acid in just the right quantity to the crude rosaniline prepared by method No. 3, and crystallizing the salt. In this case, or when the variation is adopted of adding the acid to the boiling solution of the base in lime or baryta water, the liquor must be kept hot by the injection of steam, and must be again filtered to separate sulphate of lime or baryta. The quantity of acid requisite is known by calculating the amount equivalent to the lime or baryta used in the boiling process.

The oxalate is prepared in precisely the same way from oxalic acid, and the lime water solution of rosaniline obtained by method No. 3, or prepared by heating a purified (recrystallized) aniline salt with lime in excess, and boiling with water under pressure.

Utilization of Fuchsine Residues.—In the preparation of fuchsine by the arsenic process, various kinds of residues and bye products are obtained, the nature of which vary with the modification of the process used. With the lime process (method No. 3) a mud is left consisting of the arseniate and arsenite of calcium,* humus-like decomposition products, violaniline, mauvaniline, &c., and mother liquors containing chrysotoluidine amongst other products. By the other two processes, an insoluble mass of hydrochlorides of violaniline, mauvaniline, &c., mixed with humus-like products, is formed, together with arsenical mother liquors.

Processes have been proposed for the recovery of arsenic from these residues, but as yet these methods have been but little employed. Thus Tabourin and Lemaire burnt the dried solid residues, leading the evolved gases through cooling pipes and chambers to condense the arsenic. The concentrated arsenical liquors were solidified by addition of quicklime, and the mass heated with carbon; the evolved arsenic vapours being burnt to white arsenic, and condensed as before. Weaker liquors were treated with milk of lime, and manganese chloride solution, to reduce arsenic acid to arsenious; the precipitated calcium arsenite was then treated as before.

Randu & Co. calcined the residues, collecting arsenious oxide in flues, &c.; or they calcined with coke dust, collecting metallic arsenic.

The arsenical liquors obtained by methods 1 and 2 are frequently evaporated until sodium chloride "salts out," for the most part in crystals. Nitric acid may then be added to oxidize arsenite present, and finally the resulting arseniate of soda crystallized, and sold to the calico-printer as "dunging salts."

Another method of treatment is to evaporate to a small bulk, and then distil with sulphuric acid. Chloride of arsenic passes over, and can be oxidized to arsenic acid by passing a current of chlorine gas through its aqueous solution.

Or again, the liquors are evaporated to a third of their volume; and as much sulphuric or hydrochloric acid added as corresponds to the neutral sodium arsenite present. Sodium sulphate or chloride crystallizes out, and from the filtrate an impure arsenic acid is regained, which may be used over again.

A better method of regaining the arsenic acid from these liquors is to add calcium chloride solution or milk of lime, or both, so as to obtain a precipitate of calcium arsenite and arseniate; this is then washed and treated with sulphuric acid, mixed with a little nitric. Sulphate of calcium is formed and free arsenic acid, the arsenious acid being reoxidized by the nitric acid. By evaporation and decanting from the gypsum deposited, a solution of arsenic acid is obtained, which may be used over again as a fuchsine-making agent. The lime or baryta mud obtained by method 3 may be directly treated in this way.

Mother liquors and weak liquors containing only a small quantity of colouring matter are most economically utilized by precipitating the colouring matter as a lake, which is then dried and sold as a pigment. Tannin, alumina, and stannic oxide form highly insoluble compounds with rosaniline, and are used for this purpose.

Manufacture of Violaniline, Mauvaniline, and Chrysotoluidine.—Though not red dyes, these bodies may be considered here, since they are obtained as bye products of the fuchsine manufacture. It usually happens that the fuchsine residue still contains rosaniline, from two to three hundredths being the usual amount with careful manufacture; but when the extraction process is not properly managed 4 or 5 per cent. may be left in the residue. The quantity of humus-like product formed depends much on the way in which the "melt" is manufactured. It seems to be formed chiefly at the expense of violaniline; if too high a temperature has been reached during the fusion (above 200° or 210° C.), other alteration or decomposition products are also formed. Thus at 213° C. rosaniline gives rise to a violet product, and violaniline and mauvaniline to dirty garnet-red bodies; the arsenic acid also oxidizes the bases somewhat further, giving rise to new coloured products. All of these are for the most part left in the residue, and alter injuriously the colour of the dyestuff thence extracted. It is practically found that, to obtain a good yield of fuchsine, the aniline oil should contain somewhat more aniline than that corresponding to the equation—

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* The latter salt usually contains but a small fraction of the total arsenic present, calcium arsenite being more soluble in water than the arseniate (Brinsmeyr).

$$C_6H_5.NH_2 + 2(C_7H_7.NH_2) - 3H_2 = C_{20}H_{19}N_3.$$

The oil that distils over during the operation invariably contains still less toluidine, being frequently almost pure aniline. It is highly probable that the reason this excess of aniline is advantageous in the production of rosaniline, is that it renders the mass more fluid, and thus promotes the reaction, and helps to establish uniformity of temperature and to diminish the production of these alteration products.

In 1866 SOPP patented a process for the extraction of colouring matters from the resinous residues of the arsenic acid process. By treating 100 parts of residue with 70 to 80 of hydrochloric acid, washing with water, and treating the residue with nitric acid, a yellow colouring matter is obtained in the nitric acid solution, capable of crystallizing on cooling, or of forming a pasty precipitate on addition of water. The hydrochloric acid solution contains a little fuchsine, which can be extracted by precipitating the acid solution with soda and boiling the precipitate with water. The undissolved portion gives a fine deep red liquor on treatment with ammonia water, to which a little soap has been added. By treatment with hydrochloric acid, on the other hand, it gives a violet-blue fluid which dyes solid tints, but not shades of any great beauty; fabrics dyed therewith and passed through a solution of permanganate of potassium acquire a fine chestnut colour. In all probability the yellow product thus obtained is chrysotoluidine. The extraction of chrysaniline from fuchsine residues by various solvents had been practised by NICHOLSON several years previously.

In PARAF'S process (1867) the crude fuchsine residues are digested with muriatic acid (16 parts to 30 of residues), in the cold until nothing more appears to be taken up. By filtering and adding lime to the filtrate, so as to render it exactly neutral, a fine red colouring matter is thrown down. The insoluble portion is then heated with hydrochloric acid, when a violet and a puce substance dissolve out; by adding lime to the solution these dyes are precipitated as before, and can be collected for sale. By acting on these precipitates with strong sulphuric acid much dyestuff is dissolved. The solutions when freed from sulphurous acid by boiling communicate shades of great purity to wool and silk; the insoluble residues are used as lakes and pigments for wall papers, &c. The arseniate and arsenite of calcium formed during the extraction are sold as dunging material. Different manufacturers frequently pursue special methods of their own for working up and utilizing their residues; numerous products thus obtained find their way into the trade under various names; thus *Cerise* is an impure rosaniline containing small quantities of other substances which modify its tint, crystallizability, &c.

The following process for extracting violaniline, mauvaniline, and chrysotoluidine from the residues is given by GIRARD and DE LAIRE (*Traité des Derivés de la Houille;* Paris, 1873):—1000 kilos. of residues are boiled with 12,500 litres of water, containing 85 kilos. of ordinary hydrochloric acid. Violaniline is left undissolved, and is separated along with the humus-like products by filtration; to the filtrate is added 125 kilos. of hydrochloric acid. A precipitate, A, is formed, weighing about 40–45 kilos., and consisting mainly of mauvaniline and rosaniline hydrochlorides. To the filtrate from this precipitate 685 kilos. of common salt are added, when from 30 to 35 kilos. of a mixture, B, of mauvaniline and rosaniline salts is thrown down. To the mother liquors of this precipitate 83 kilos. of sodium carbonate at 59·8 per cent. Na_2O (*i.e.*, almost perfectly pure) are added; from 205 to 210 kilos. of precipitate C is obtained, consisting chiefly of rosaniline salt, with a little chrysotoluidine. Finally, 37½ kilos. of sodium carbonate are added to this filtrate, and a precipitate, D, weighing about 37 to 40 kilos., and consisting of little but chrysotoluidine is obtained.

Precipitate A is dried and left in contact for 24 hours with twice its weight of hydrochloric acid and 10 times its weight of water, the whole being stirred from time to time. After filtration the filtrate is precipitated by carbonate of soda, when, for every 100 kilos. of dry precipitate A, 32 kilos. of precipitate like A is thrown down; this is treated over again as A. The residue on the filter is treated twice successively with 1000 litres of a mixture of equal bulks water and hydrochloric acid, and a precipitate obtained from the liquid by adding 500 litres of cold water: in each case about 4 kilos. of nearly pure mauvaniline hydrochloride are thrown down. By addition of carbonate of soda to the mother liquors about 4 kilos. more mauvaniline are obtained, and by further saturation 4 kilos. of impure fuchsine. For each 100 parts of dry matter, A, there are obtained—Impure fuchsine, 4 per cent.; matter resembling A, 32 per cent.; mauvaniline, 12 per cent.; residue insoluble in acid and loss, 52 per cent. The mauvaniline hydrochloride precipitate is next dried and heated with benzol to dissolve out a little resinous matter, and is then cohobated with alcoholic potash; the free mauvaniline is then converted into any required salt, *e.g.* (such as the acetate, which yields an aqueous solution dyeing a magnificent mauve).

Precipitate B is either sold as "violet fuchsine," and used directly for dyeing, or is treated as A, but with acid containing only one-fifth of hydrochloric acid instead of one-half; it is thus separated into mauvaniline and rosaniline.

Precipitate C is dissolved in 2000 litres of water, containing 5 kilos. of hydrochloric acid per 100 kilos. of precipitate; the solution is filtered into a crystallizing pan, when from 25 to 30 kilos. of pure rosaniline hydrochloride are deposited. The mother liquors contain the chrysotoluidine and are used over again in the same way; after which common salt is added, and a precipitate resembling C is obtained (and treated as C), the brine yielding a precipitate of impure chrysotoluidine by addition of carbonate of soda. This is treated with precipitate D.

Precipitate D.—100 kilos. of this precipitate are dissolved in 2500 litres of lime water, containing a little lime in suspension. After boiling for three or four hours the liquor is filtered into a vat of dilute hydrochloric acid (sulphuric acid may be employed, but not so advantageously). On cooling, crystals of "yellow fuchsine," or rosaniline mixed with a little chrysotoluidine, are deposited. The portion insoluble in lime water is heated in an enamelled digester with a little water, and enough hydrochloric acid to neutralize the lime; the chrysotoluidine melts, agglomerates, floats on the liquid, and is removed by skimming. To separate the last traces of rosaniline, 100 kilos. of skimmings are dissolved in 2000 litres of boiling water, acidulated with 100 kilos. of hydrochloric acid; 10 kilos. of zinc are then added, and the whole boiled for eight hours. The rosaniline is thus reduced to leucaniline, which is not so easily precipitated by common salt; the filtered liquor is treated with salt (20 kilos.) and a little sodium carbonate to neutralize the acid, when 80 kilos. of amorphous chrysotoluidine are thrown down. This is again dissolved in 2000 litres of slightly acidulated water, filtered, and precipitated with 50 kilos. of caustic soda ley of 12° B. The precipitate is washed with cold water, and boiled for two hours with 2000 litres of hot water and 8 kilos. of sulphuric acid; the cooled liquor is partially precipitated with carbonate of soda. 20 kilos. of chestnut-brown-yellow precipitate are obtained. By further addition of common salt, 25 kilos. of pure yellow chrysotoluidine are thrown down; which is transformed into sulphate by treating with exactly the necessary quantity of sulphuric acid. 100 kilos. of dry precipitate D yield a chestnut-yellow dyestuff suitable for leather, &c., 20 per cent.; chrysotoluidine, 25 per cent.; the losses, chiefly as brown matters, are 55 per cent.

Rosaniline Mother liquors containing chrysotoluidine are worked up as follows, in order to separate it:—If acid, they are neutralized by carbonate of soda; common salt is then added, and the precipitated colouring matters are treated with zinc and hydrochloric acid as above, to convert rosaniline into leucaniline. Jacobsen states that "aniline orange" (chrysotoluidine?) may be obtained from the mother liquors of azaleine (mercury nitrate process) by precipitating the violet and brown dyestuffs by common salt, and crystallizing the filtrate.

Another process for the extraction of violaniline, mauvaniline, and chrysotoluidine from fuchsine residues, is to heat these (previously dried) with aniline to 100° C.; the bases are thus dissolved, and can be separated by filtration from the humus-like bye products. On neutralizing the aniline with an acid, violaniline is precipitated, the other two being retained in solution by the aniline salt formed. By means of common salt a precipitate of impure mauvaniline is thrown down, whilst the chrysotoluidine remains in solution, and can be separated by adding an alkali and distilling off the liberated aniline by means of steam.

It appears probable that considerable quantities of aniline bases might be obtained from fuchsine residues by simple dry distillation. By thus treating a resinous product of similar character, obtained as a bye product in the preparation of Hofmann's violet, and known accordingly as "Hofmann's gum," Spiller has recently obtained a large yield of methyl aniline or ethyl aniline, accordingly as ethyl or methyl violets were being made in the process of which the gum was a bye product. The gum is kept fused for some time, so as to drive off nearly the whole of the inclosed water, and is then heated in an iron still over a coke fire, either with or without the addition of roughly powdered charcoal. A mixture of pulverized iron borings, kaolin, and syrupy silicate of soda, forms an excellent lute for fixing on the head of the still, as it withstands a high temperature without softening. Small quantities of water and ammonia pass over together with a quantity of oily distillate, in quantity equal to half the weight of gum used. This distillate consists almost wholly of methyl- (or ethyl-) aniline. (Proc. Roy. Soc. xxi. 204.)

Manufacture of Rosatoluidine (*Rouge de toluène*).—Coupier patented in 1866 the following processes:—I. Coupier's toluidine (boiling at 198° to 202° C., and made from pure toluene separated by Coupier's fractionation process, *vide* Benzol) is mixed with 160 per cent. of its weight of arsenic acid containing 75 per cent of "real acid," and 25 per cent. of its weight of hydrochloric acid of commerce. The whole is quickly heated to 150° to 160° C. for three hours in a cast-iron still until the mass thickens, so that it can be drawn out into long brittle strings. The presence of hydrochloric acid is a new feature in this process, and also the low temperature employed; the mass is less likely to stick to the pot and be burnt, on account of the greater fluidity due to the excess of aqueous acid.

Process II.—67 parts Coupier's toluidine, 95 parts nitrotoluidine, from which the above is made; 65 parts commercial hydrochloric acid; 7 parts ferrous chloride, or its equivalent of ferric chloride. The above mixture is heated for three hours to 190° C. The addition of the iron salt greatly facilitates the reaction. The extraction of the colouring matter from the products is effected in the ordinary way adopted in the case of fuchsine.

Rosenstiehl found that the yield of crystallizable red dyestuff, from a mixture of 2 parts paratoluidine and 1 metatoluidine, by treatment with arsenic acid, was 39 per cent.; whilst a mixture of 2 parts metatoluidine and 1 of aniline gave the enormous yield of 50 per cent.—the yield from various mixtures of aniline and paratoluidine being in no case greater than 30 per cent. The maximum yield is obtained when equal weights of the two alkaloids are used. In all these cases the weight of bases regained by distillation is subtracted from the amount originally employed.

Coupier states that his toluidine, containing 95 per cent. of a mixture of para- and meta- toluidine, furnishes from 45 to 50 per cent. of crystallizable red, whilst any admixture lessens the yield. Roussille finds that the tinctorial power of rosatoluidine is 140, that of rosaniline being taken as 100 (*Bull. Soc. Chim.* Paris, [2] iv. 312).

Toluidine red gives to vegetable and animal fabrics a shade rather more blue than that communicated by fuchsine.

Xylidine Red.—A product has been introduced by Coupier under this name, derived from xylidine by the same processes as those by which rouge de toluène is got from Coupier's toluidine. The following formulæ are given by Coupier:—Xylidine, 100 parts; arsenic acid at 75 per cent., 140 parts; commercial hydrochloric acid, 20 parts: heat for three hours to 130° to 140° C. Or heat to 200° C. xylidine, 75 parts; and of the corresponding nitroxylidine, 105 parts. This red dyes animal and vegetable fibres a more violet shade than fuchsine and rosatoluidine. Of the composition of this red, and its relation to rosaniline and the other red dyes, nothing is known; but without doubt its composition is analogous to that of rosaniline. These results are entirely different from those of Hofmann, who found that xylidine alone, or mixed with toluidine, gave no red; but that a fine red could be formed from a mixture of aniline and xylidine by the action of arsenic acid. The red derived from aniline and xylidine is not necessarily identical with the "xylidine red" or "rosaxylidine" of Coupier.

Ulrich's Scarlet.—Four parts of rosaniline acetate are dissolved in hot water, and mixed with a solution containing 3 parts of lead nitrate; the whole is then evaporated to dryness, and heated to 150° to 200° C. until the whole mass is violet. After cooling the mass is boiled for a long time with very dilute sulphuric acid; an excess of alkali is added to the hot liquor, and the whole filtered whilst hot. The new colouring matter is contained in the filtrate, and may be precipitated by addition of salt. Perkin's mauve may also be used, and peroxide of hydrogen and other oxidizing agents can be employed with the same result: it is therefore probable that this scarlet is closely allied to Geranosine.

Geranosine.—Luthringer gives the following process for the preparation of this dyestuff:—4½ kilos. of barium or calcium nitrite or dioxide are disseminated through 25 litres of water, and 10 kilos. of strong sulphuric acid (at 66° B) are added; to this liquor is added a solution of 1 kilo. of any rosaniline salt dissolved in 1000 litres of water, and at a temperature of 45° C.; the mixture becomes citron yellow and then colourless. After filtration the temperature is gradually raised to 100° C. to separate barium sulphate, when an intense red tint is developed; after two minutes' boiling the liquor is cooled, and the dyestuff salted out and collected on filters.

This dye is not decolorized by ammonia (alkalies greatly weaken or destroy the fuchsine tint by setting free the nearly colourless basis); it is soluble either in water or alcohol.

Saffranine.—Mène gives the following process:—Aniline nitrite is prepared by passing into aniline the vapours evolved from a mixture of starch and nitric acid. The product is washed two or three times with water, and then heated with half its weight of arsenic acid to 80° to 120° C. for five minutes; the product is poured into boiling water, and lime added until the acid is neutralized. The liquor is then filtered through flannel, and the colouring matter salted out from the clear red liquor by means of common salt (5 parts to 1 of aniline nitrite). The pasty mass is then drained, and pressed to remove the brine. In preparing the aniline nitrite care must be taken that the temperature does not rise too high, otherwise the aniline will be carbonized; the delivery pipe should dip pretty deeply into the oil, and the containing vessel should be frequently agitated and cooled by water. An excess of nitrous acid must also be avoided. Besides arsenic acid, other oxidizing agents may be employed, viz.: chromic acid, a mixture of sulphuric acid and potassium dichromate, stannic chloride, &c. Of these potassium dichromate seems to be the most suitable. Simultaneously with saffranine, a violet colouring matter is formed; this is precipitated by the alkaline solution added, and is then separated.

Manufacture of Bleu de Lyons or Aniline Blue.—The preparation of good aniline blue from fuchsine and aniline oils, by simply heating the ingredients together, is an operation of some delicacy, the result depending to a great extent upon circumstances which at first sight would hardly seem to be important. The original process patented by Girard and De Laire was as follows:—Purified fuchsine is mixed with its own weight of aniline oil, and the mixture heated to a temperature as near 165° C. as possible (between 155° and 185°) for five or six hours: a violet product is obtained, 1 part of which is boiled with 10 to 12 parts of hydrochloric acid diluted with the same quantity of water; unaltered fuchsine and aniline are dissolved out, and a violet dyestuff is left. By further boiling this product with hydrochloric acid (10 of acid to 100 of water) several times successively, a pure blue colouring matter is left, which is dissolved in acetic acid, alcohol, or wood spirit, and the solution used for dying.

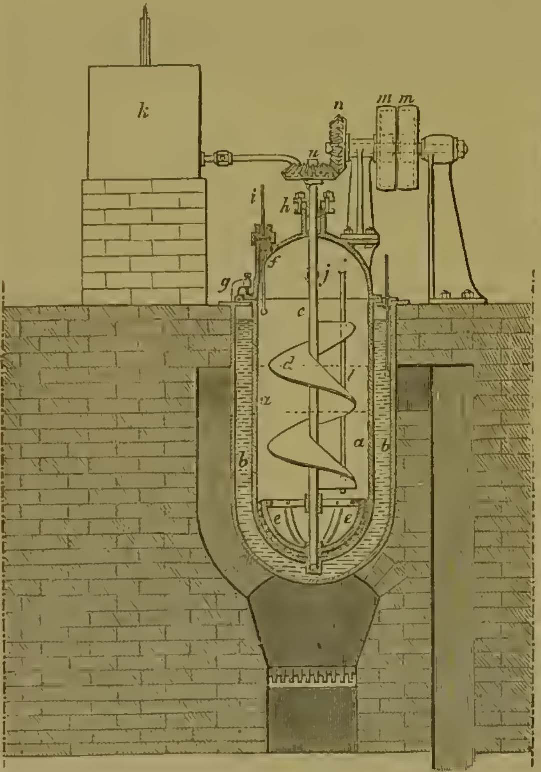
According to Reimann, it is possible to get every shade of violet in this way, but no true blue, unless the rosaniline salt of an organic acid be used, or what amounts to the same thing, unless an organic sodium or other metallic salt be added to the mass; moreover, a much larger proportion of aniline is desirable.

Levinstein's process is worked as follows:—Three parts of aniline and one of magenta are heated to 180° C. for three hours, when the red becomes violet; half a part of potassium acetate is then added, and the temperature raised to 190° C. for one hour and a half, or until a sample taken out appears of a greenish blue hue against a white background; the mass is then treated with hydrochloric acid as before. The blue thus obtained has a faint greenish tinge.

Passavant's method was to heat 4 parts magenta, 8 of aniline, and 2 of sodium acetate to 200° C. for two hours, the temperature being then raised to 250° C. When the required shade is attained the semi-fluid mass is poured into a mixture of 4 parts alcohol and 12 hydrochloric acid; the brownish cooled mass is then powdered and boiled for fifteen minutes with dilute sulphuric acid, and filtered. The residue is boiled twice successively with hydrochloric acid to eliminate the last traces of red tinge.

Bardy describes the following manufacturing process:—Rosaniline hydrochloride, 100 parts, sodium acetate, 30 parts, crystallized potassium acetate, 10 parts. The first two ingredients are mixed and evaporated to dryness; the potassium acetate and some free aniline are then added, and the whole heated to 175° C. till a pure blue tint is developed. The product is then treated with one and a half times its weight of strong hydrochloric acid; this leaves the blue undissolved. The insoluble substance is collected, washed with water, and treated with five times its weight of caustic soda ley at 32° C. After twenty minutes boiling 15 parts of boiling water are added and the whole filtered. The free base thus produced is washed with lukewarm alcohol to remove mono- and di-phenyl rosaniline, and then treated with sulphuric acid.

Fig. 8.



The hydrochloric acid solution yields a precipitate of diphenyl rosaniline in dilution with water equal to $1\frac{1}{3}$ parts per 1 part of crude blue; on further adding 23 parts of water, monophenyl rosaniline is thrown down. The mother liquors are saturated with lime and mixed with acetate and chloride of sodium to recover rosaniline hydrochloride.

The process as carried out at the present day is thus described by Girard and De Laire:—An enamelled retort holding about 20 litres is heated in a paraffin bath, a cohobator and distilling worm are attached to the top (which is movable), and an agitator is provided.

Fig. 6 shows the apparatus in section through the axis; *a*, enamelled cast-iron retort; *b*, paraffin bath; *c*, *d*, *e*, agitator; the top part (*d*) helical, the lowest portion (*e*) provided with curved arms furnished with projections almost touching the bottom of the retort to prevent adhesion of materials; *f*, movable top: *g*, flange and screw nuts; *h*, packing box of agitator axle; *i*, thermometer; *j*, orifice with which the cohobator and condenser are connected, either being shut off at will by suitable taps; *k*, cohobation worm (the distilling worm is below on the opposite side of the retort); *l*, tube for blowing steam through (a hollow agitator axle may also be used); *m*, *n*, pulley and cog-wheels.

Five kilos. of acetate of rosaniline and 15 of aniline are introduced into the retort, which is then heated to 170° C. for about two hours. Samples are taken from time to time by introducing acid through a tubulation provided for the purpose. A drop is placed on a white porcelain dish, and a drop of alcohol and acetic acid added. When the operation is complete a pure blue colour should result; if a strongly marked red margin is perceptible, the action has not gone on long enough; if the aureole is not of a bright rose, but is brown or grey, the operation has been carried on too long. By practice the workman is able to hit the exact point when the product is not quite liquid, but is perfectly homogeneous, the blue being wholly dissolved in the excess of aniline. This excess (three and one-fourth times the theoretical amount) is desirable, as it enables a constant temperature to be kept up by rendering the whole tolerably fluid, and thus greatly promotes the action and improves the products. A number of these retorts may be conveniently connected with one condensing worm and worked together.

Direct Blue.—To prepare this dye all that is requisite is to free the mass from aniline. This is readily accomplished by blowing steam through, or the colour may be dissolved out by acids.

Purified Blue.—The crude product is diluted with alcohol (methylated spirit or wood spirit), and run in a thin stream into water acidulated with hydrochloric, acetic, or sulphuric acid. The blue is precipitated as a fine powder, most of the brownish impurities and bye products being retained in solution by the slightly alcoholic solution of aniline salts thus produced. The blue precipitate is then washed with boiling slightly acidulated water, and is fit for the market.

Night Blues (Bleus lumières) are prepared by washing the purified blues with warm alcohol, and dissolving in boiling alcohol. To the filtered liquid is added alcoholic ammonia or soda, when triphenyl rosaniline is precipitated in a state of considerable purity. The precipitate is then washed with boiling water and dissolved in the particular acid the salt of which is required. Bleus lumières are also sometimes prepared by boiling purified blue with a mixture of alcohol and hydrochloric acid, and allowing the whole to cool. Triphenyl rosaniline hydrochloride is less readily taken up than the impurities, and is more easily deposited. The undissolved substance is treated thus several times successively, and is then obtained pure enough for sale. Both of these processes require the use of much alcohol, and in the latter a considerable loss is occasioned through the formation of ethyl chloride. Aniline oils are therefore often used instead of alcohol as solvents. The mother liquors from these purifications are preserved and distilled with lime; alcohol first passes, then aniline and water. Unaltered fuchsine and yellow, brown, violet, &c., bye products are left in the still (together with benzoate of lime, if benzoic acid were used in the preparation of the aniline blue).

Slight modifications are made in these processes for special qualities of dyestuffs, indicated in commerce by the marks B, B B, B B B, and B B B B respectively.

Blue B.—To make a dye of this quality 2 kilos. of pure rosaniline, 3 of aniline boiling at 182° to 185° C. and free from hydrocarbons, and 270 grammes of glacial acetic acid, or of benzoic acid, are heated together for two hours, the temperature not rising above 180° C. Benzoic acid gives slightly greener shades than acetic acid, and the dyes are suitable for silk, whilst wools require dyes made with acetic acid. The aniline oil is used as pure as possible, the tritoluyl rosaniline being formed with greater difficulty, and requiring more numerous treatments to prepare purified blue than triphenyl rosaniline. The aniline that distils over during the preparation of fuchsine answers well. When the shade given by a sample treated with alcohol and acetic acid is a pure blue, with no trace of violet or grey, the contents of the retort are run into a vat (of wood or enamelled cast-iron) containing 10 kilos. of strong hydrochloric acid, with continual agitation; the mass is filtered and pressed, and well boiled up by steam with water acidulated with hydrochloric acid, until the blue is reduced to a fine powder. This is then filtered and pressed: 3500 grammes of dry blue are thus obtained. If the acetate or sulphate is required, the corresponding acid must be used in lieu of hydrochloric.

Blue B B.—The proportions for this shade are different, viz.: pure rosaniline, 2 kilos.; pure aniline, 5 litres; and glacial acetic acid, or benzoic acid, 270 grammes. The whole is treated as for the quality B. The blue is heated in a cohobator, with alcohol and benzol (1 part of blue purified up to the stage of blue B, 1¼ part concentrated alcohol, and 5 parts highly rectified benzol). After boiling one hour the whole is filtered (through closed-in filters), pressed, and dried; the alcohol and benzol are distilled off from the mother liquors and used again, an inferior blue being left behind. A mixture of alcohol and aniline is sometimes used, in which case the blue is washed finally with an acid to remove adhering aniline.

Instead of the above process, the contents of the retort may be emptied into an enamelled iron pot surrounded with water, so as to cool them quickly, and 6 to 8 kilos. of strong alcohol added, with agitation. The homogeneous mass is then heated until the alcohol just begins to boil, cooled, and precipitated by the addition of from 6 to 12 kilos. of strong hydrochloric acid added, with continual stirring; heat is evolved from the formation of aniline hydrochloride. To obtain a uniform product the precipitate should be filtered off at a temperature of 45° C., pressed, thoroughly washed with a large bulk of water, and dried; 1320 grammes of blue B B should be obtained.

Blues B B B and B B B B.—1 kilo. of dry pulverized blue B B is boiled with 26 kilos. of alcohol in a cohobator provided with an agitator; 2 kilos. of 20 per cent. alcoholic caustic soda solution are then added. The whole is well mixed and then filtered through a closed-in filter, the presence of the hot alcohol greatly assisting the operation (the cohobator tap being turned off): 280 grammes of strong hydrochloric acid are then mixed with the alcoholic liquor while yet warm, and the whole is allowed to stand two days. The crystalline precipitate is then drained and pressed—this is blue B B B; by repeating the treatment blue B B B B is obtained. One kilo. of blue B B gives 680 to 690 grammes of pure dry blue B B B; an inferior blue is obtained from the matter left in the filter, and the alcoholic mother liquors, by dilution with water. The aqueous acid alcohol is rectified over lime and recovered.

Instead of pure alcohol a mixture of alcohol and aniline may be used with advantage: 1 kilo. of pure dry blue B B, 1 of alcohol, and 2 of aniline, are well mixed while heating on the water bath; the viscid homogeneous mass is then poured into 25 kilos. of alcohol, and heated to boiling; the alcoholic soda is then added, and the liquor filtered and precipitated with a slight excess of hydrochloric acid. After 48 hours the precipitate is filtered off, washed with boiling water slightly acidulated, and dried; about 800 grammes of blue B B B are thus obtained, and a proportionally less quantity of inferior blue.

According to REIMANN it is impossible to get a pure full-blue from any simple mixture of aniline and magenta (rosaniline hydrochloride?). By heating together 100 parts of magenta, 100 of aniline oil, and 25 of sodium acetate, at 170° to 180° C. in an oil bath, a red violet is easily formed, and by further heating, violets and blue violets. To prepare these latter, and to transform them into a full blue or greenish blue, benzoic acid, benzoate of potassium or sodium, formate of sodium, stearic acid, stearate of sodium (common tallow soap), or some similar substance must be added. Thus, to get a blue violet 1 part of aniline, 3 of magenta, ¾ of sodium acetate, and ⅔ part of soap are used, the same substances being used for blue with ¾ part of soap; the soap being added in small quantities when the other materials have acquired a reddish blue tint, and well incorporated. This product must be quickly removed when the proper shade is acquired, otherwise it becomes overheated and damaged. The mass is then treated with dilute hydrochloric acid to remove aniline and fuchsine, and well washed with water, the stearic acid set free by the action of the acid on the soap being subsequently dissolved out by benzol: 100 parts of magenta give 150 of blue when the operation is successful.

The discrepancy between Reimann's statements and those of Girard and De Laire probably arises from the use of larger quantities of aniline by the latter, and the choice of aniline oils almost wholly free from higher homologues. Toluidine acts on fuchsine somewhat less readily than does pure aniline, and the resulting "toluidine blue" differs in some respects, notably in solubility and shade of colour; it is also much less easily purified, and hence is but rarely made. A patent for the production of this body was taken out by Collin in 1862, the process specified being the heating of equal weights of a rosaniline salt and of crystallized toluidine (paratoluidine) to between 150° and 180° for five or six hours. The unaltered toluidine and rosaniline are dissolved out from the crude mass by boiling with eight to ten times its weight of dilute hydrochloric acid. Clark patented a precisely similar process in 1864.

Coupier has prepared a blue from rosatoluidine by the action of aniline. A similar blue is also obtained by the action of toluidine (mixture of para- and meta-toluidine) on rosatoluidine, and according to him it possesses great beauty, and is specially easy to purify.

Manufacture of Soluble Blue (*Nicholson's Blue, Alkali Blue*).—The soluble blue manufactured at the present day differs slightly in composition from that prepared some years ago, being essentially the sodium salt of the mono-sulphonic acid of triphenyl rosaniline $C_{20}H_{15}(C_6H_5)_3(SO_3Na)N_3$ or $C_{20}H_{16}(C_6H_5)_2(C_6H_4SO_3Na)N_3$, whereas the older dyes were the salts (ammonium, sodium, &c.) of the corresponding di- and tri- sulphonic acids. The mono-product is somewhat less soluble in water than the others, but it yields much more stable shades.

The higher sulpho-derivatives were prepared thus:—Triphenyl rosaniline sulphate was heated by steam in a jacketed vessel to 130° to 140° C., with from 4 to 10 times its weight of strong sulphuric acid; the mass was then cooled and poured by small quantities into 8 to 10 times its weight of water, the precipitate being then collected on a filter, drained, and pressed (or dried in a centrifugal machine). It was then warmed with a slight excess of ammonia in an enamelled pan: the coloured salt rising to the surface as a golden mass, when it was skimmed off, dried, and powdered. In his original patent (June, 1862), Nicholson employed 10 parts of acid to 1 of blue, and heated for about an hour and a half to 150° C.; but it was soon found that a rather lower temperature and a smaller proportion of acid gave better results. A purified product, known as *Soluble Night Blue* (bleu lumière soluble), is obtained from this substance by solution in alcohol, when ammonium sulphate and other impurities are left undissolved.

The process now employed is virtually the same, with the difference that less acid is used (twice to three times the weight) and the temperature is lower, not exceeding 100° C. at the most. Thus a kilo. of purified blue B B, in fine powder, may be added to 3 litres of strong sulphuric acid on the water bath in small portions at a time, until a drop thrown into water gives a blue precipitate which does not colour water when washed by decantation several times, but is nevertheless wholly soluble in caustic soda or ammonia. The whole is then poured with continual stirring into wooden vats containing 30 litres of water; after twenty-four hours the precipitate is collected, drained, and heated on the water bath or by steam, with the exact quantity of caustic soda required. The product is dried on enamelled iron plates in a hot chamber, pulverized, and mixed with 20 per cent. of its weight of soda crystals.

Or two parts of acid and one of blue B B are treated at as low a temperature as possible until rendered just soluble; the product being precipitated by water, washed, pressed, and dried.

Vogel proposed to heat triphenyl rosaniline with 8 parts of fuming sulphuric acid to 130° for six hours: higher substitution acids than the mono-sulphuric acid are thus produced.

Leonhardt prepares a very finely divided blue, by dissolving the soluble blue in alcohol, and then precipitating with water.

The alkaline salts of soluble blue are almost colourless; to develop and fix the colours an organic acid (such as acetic) is preferable to a mineral one.

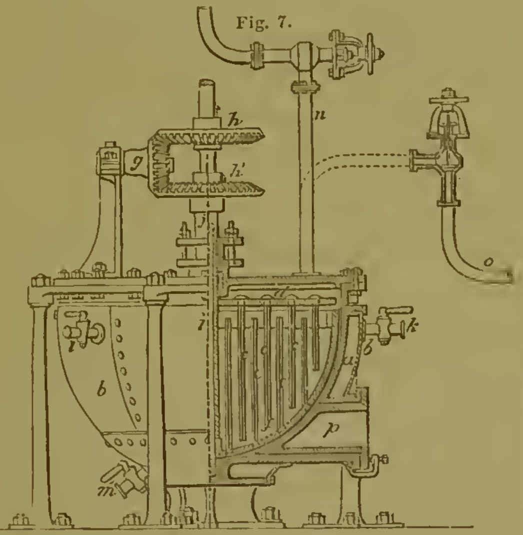
Manufacture of Diphenylamine Blue.—Although any fuchsine-making material will transform commercial diphenylamine (true diphenylamine mixed with higher homologues) into blue, yet the so-called carbon sesquichloride (C_2Cl_6) gives the best results, both as respects yield and quickness of working. Brinmeyr has, however, obtained the same product by simply heating diphenylamine and oxalic acid to 110° to 120° C. for some hours.

Girard and De Laire thus describe their process; the operation is effected in enamelled iron retorts provided with agitators, and heated in an oil bath, their capacity being about 50 litres:—12 kilos. of sesquichloride of carbon, and 10 of commercial diphenylamine, are introduced and gently heated; at 160° C. the reaction commences, and at 180° C. it goes on steadily; a higher temperature risks the formation of a violet product in lieu of a blue one. So-called protochloride of carbon (C_2Cl_4) distils over throughout the operation, and is collected in a graduated vessel; a quantity almost exactly corresponding to the amount of sesquichloride used should be obtained. A sample is drawn from the retort with a rod from time to time; when this (after cooling) is of a copper red colour, does not soil the fingers, is dry and friable, and dissolves in alcohol to an intensely blue liquor. the calculated quantity of protochloride of carbon having been likewise obtained, the operation is finished. This usually takes from three to four hours. The mass is then poured out upon iron cooling plates, and purified by one or other of the following processes:

No. 1. One part of crude blue is dissolved in two of aniline, at a temperature not above 100° C., and the solution poured in small quantities, with continual stirring, into 10 parts of benzol; a closed vessel should be used to avoid loss by evaporation, two hours at least being required for this operation. The precipitated blue is collected on calico filters, drained, pressed, and treated in the same way over again; the drained (but not pressed) precipitate thus obtained is washed with 5 parts of benzol, with agitation for two hours, The powder is then collected on a filter, dissolved by heat in 2 parts of aniline, and precipitated by 4 parts of ordinary hydrochloric or acetic acid. The precipitate is washed with a little boiling water (the runnings being saved to regain the aniline hydrochloride) and then with a large bulk of hot water: care must be taken not to use calcareous water.

No. 2. The crude blue is boiled with an alkaline lye in an apparatus such as that denoted in Fig. 7, in which *a* is a hemispherical vat, surrounded by a steam jacket, *b*; *c*, *c*, are the vanes of the upper agitator, *d*; and *e*, *e*, the vanes of the lower agitator, *f*; *d* and *f* are two agitators having their respective motions in opposite directions, and working after the fashion of a Carr's disintegrator; *g*, *h*, and *h′*, the cogwheels for giving these motions; *i* is the *solid* axle of lower agitator; and *j*, the *hollow* axle of the upper agitator; *k*, *l*, taps at the entrance and exit steam-pipes; *m*, a tap admitting steam into the interior; *n*, the connection with cohobator; *o*, the connection with distilling worm (neither of these connections are necessary for aqueous solutions); *p*, the discharging orifice. The base set free is pressed, dried, pulverized, and exhausted with petroleum hydrocarbons boiling at 70° to 100° C. in closed vessels. The residue is then boiled with water (to expel the hydrocarbons) dried, pulverized, and then dissolved in warm alcohol, filtered, and precipitated by 10 per cent. of strong hydrochloric acid; some impure hydrochloride of rosaniline is thus thrown down. The liquor containing the colouring matter is exactly neutralized with soda and subjected to distillation to obtain a crystalline blue, which is then washed with boiling water to get rid of alkaline salts.

Fig. 7.



No. 3. The crude blue is dissolved at a temperature not higher than 60° C. in 10 parts of strong sulphuric acid. When complete solution is effected, 30 parts of water are added; the blue is precipitated, whilst a violet bye product remains for the most part in solution. The liquor is filtered through asbestos or glass powder, and washed, first with dilute acid, such as the mother liquors themselves, and finally with pure water, till no longer acid: the mass is then pressed, dried, and purified either by benzol, or petroleum and alcohol, as in processes Nos. 1 and 2.

The violet residues from these processes are collected and subjected to distillation, to regain diphenylamine.

No. 4. The petroleum treatment is adopted, as in No. 2, the blue is then dissolved in acetic acid, and the resulting acetate in a mixture of 4 parts aniline and 10 of alcohol; the liquor is filtered in covered vessels, and the base set free by alcoholic soda. Finally, the liquor is filtered and precipitated by hydrochloric acid. A chemically pure product is thus obtained.

The acid-alcoholic aniline solutions are diluted with water, and then rectified over lime.

Preparation of Mulhouse Blue.—Gros Renaud and Schœffer boil together:—White gum lac, 50 grms.; sodium carbonate, 18 grms.; solution of azaleine (rosaniline nitrate), 50 grms.; water, 1 litre, for one hour, water being added from time to time to supply loss by evaporation. The azaleine solution referred to is prepared by dissolving 125 grms. solid azaleine in 1 litre of a mixture of equal volumes of water and alcohol. The resulting liquor possesses the same tint as that of an ammoniacal solution of a copper salt. A violet product (*Violet de Mulhouse*) may be obtained by using more of the azaleine solution; this is probably only a mixture of the blue with unaltered azaleine.

Preparation of Aldehyde Blue.—This is prepared in the way described under aldehyde green. Many attempts have been made to utilize this dye, especially for worsted, &c.; but its want of permanence is too great.

Preparation of Azuline.—Marnas prepares this blue colour by heating to the boiling point a mixture of 5 parts of coralline (*péonine*, the product of the action of ammonia on rosolic acid, *vide* article Carbolic Acid), and aniline 6 to 8 parts, until nearly all is transformed into a blue colouring matter, which is purified by successive washings, first with water acidulated with hydrochloric or sulphuric acid; secondly, with hot oils from coal tar; next, with dilute caustic alkali; and finally, with water slightly acidulated. The product is a reddish powder with a golden lustre, soluble in alcohol and methylated spirits, which solution can be used for dyeing. An analogous blue colour is producible by the action of naphthylamine on rosolic acid.

Manufacture of Azurine.—Calvert, Lowe, and Clift printed cotton goods with this tint in the following way:—A liquor is prepared from chlorate of potassium, 1 part, and water, 40 parts. The goods are rinsed in this liquor, dried, and then printed with a 1 per cent. solution of aniline tartrate or hydrochloride, thickened with starch. Or, the following mixture is taken for printing on the cotton goods without any previous treatment:—1 part of aniline, 2 of tartaric acid, 60 of starch, and 1 of potassium chlorate. The potassium chlorate is dissolved in the hot starch paste, and after cooling the aniline bitartrate solution is added. In this way a *green* tint, "Emeraldine," is communicated. When the goods thus printed are immersed in a bath of 1000 parts of water and 25 of soap, or of 1000 parts of water and 6 of caustic soda, or of 1000 parts of water and 6 of potassium dichromate, this green tint becomes blue (Azurine).

BLUMER-ZWEIFEL uses the following formula for Emeraldine —Water, 1000 parts; starch, 100; potassium chlorate, 4; sulphate of iron, 3 to 4; sal ammoniac, 10: a salt of aniline, 60. The water is boiled and the starch is made into paste with it; the chlorate of potassium, sulphate of iron, and sal ammoniac are dissolved in the hot liquor; after cooling the aniline salt is added. Another recipe is as follows:—15 parts of nitric acid, 6 to 8 of water, and 10 of aniline. To this add sufficient starch or British gum to thicken, together with 10 parts of sugar, and 4 to 8 of potassium or sodium chlorate. The Emeraldine is developed by exposing the goods to the air for from twenty-four to thirty-six hours after printing, and the blue tint is then given by one of the above baths for that purpose.

Manufacture of Mauve.—The following description of this manufacture is given by PERKIN (*Chem. Soc. Journal*, 1861):—Solutions of equivalent proportions of aniline sulphate and potassium dichromate are mixed and allowed to stand; the whole is then filtered and washed with water until free from potassium sulphate. The precipitate is dried and digested several times successively with coal tar naphtha, until all resinous matter is separated, and the naphtha is no longer coloured brown. After this it is repeatedly boiled with alcohol to extract the colouring matter. The alcoholic solution when distilled, leaves the dyestuff at the bottom of the retort as a beautiful bronze-coloured substance.

The product as thus prepared, though suitable for practical purposes, is not chemically pure: if required pure, it is best to boil it in a large quantity of water, to filter off the coloured solution thus obtained, and to precipitate the colouring matter by addition of an alkali. The precipitate should then be washed with water till free from alkali, and then dried. The residue is dissolved in absolute alcohol, and the solution evaporated on the water bath.

SCHEURER KESTNER describes the following industrial process:—Mix commercial aniline, 1 kilo.; cold saturated aqueous solution of potassium dichromate, 800 to 1200 grains; sulphuric acid at 66° B, 500 grammes. Wash the precipitate first with cold and then with boiling water, to which 1 to 2 per cent. of acetic acid is added; evaporate the acid filtrate, filter, and add caustic soda till no more precipitate is thrown down; collect the precipitate and wash with a dilute alkaline liquor, whereby a red colouring matter is removed; then wash with water till this filtrate is colourless; drain the resulting paste thoroughly. The dyestuff may be sold thus, or the paste may be purified by solution in boiling water and precipitation by alkali, or by solution in alcohol and evaporation to dryness.

SCHLUMBERGER describes the following process as worked by GEIGY & Co., of Basle:—Mix in a vat holding 400 litres, and heated by a steam tube, 60 litres of water, 4 kilos. of aniline, and 2·12 kilos. of sulphuric acid at 66° B. This mixture is boiled, and then allowed to cool. When cold they add in a thin stream, and with continual stirring, a cold solution of 6·36 kilos. of potassium dichromate and 40 litres of water. The mixture is agitated at intervals for two days, and hot water is then added till the vat is full. After settling, the precipitate is washed by decantation and filtration, with diluted sulphuric acid at 2° B, lastly, the filtrate is washed with water, till it is no longer yellow.

From this product the colouring matter is dissolved out by boiling by steam for two hours in wooden vats holding about 400 litres. The filtered liquors are precipitated with caustic soda and common salt; the precipitate is collected, slightly washed to remove excess of alkali; and the drained paste is then treated with 5 per cent. of acetic acid, boiled with water, and the filtered liquid again precipitated with soda. This process is laborious and costly, four days being required for the aqueous extraction. Instead of water, spirit of wine may be used. The crude washed precipitate is dried on metal plates, and then boiled with alcohol in a copper extracting vessel, consisting of two boilers, each heated by steam. The upper one has a perforated false bottom, on which is a calico filter cloth, above this is a fine sieve, and above this again, straw. The powder to be extracted is laid on the straw; alcohol is poured on it, and the whole heated by the steam-worm which lies under the false bottom; the vapours are condensed by a worm tube, and flow back on to the crude colouring matter. The lower boiler is so arranged as to receive the alcoholic solution which percolates through the filtering bed in the upper vessel; it is provided with a steam jacket, so that the alcohol can be distilled off. The condensed alcohol is made to drop back over the matter to be exhausted, and thus the alcohol is used over and over again until no more colour is dissolved out. On distilling off the whole of the alcohol, a pasty mass of colour is left; this is dissolved in boiling water, filtered, and precipitated with soda. According to SCHLUMBERGER, less dyestuff is obtainable by this process in summer than in winter, owing to the greater difficulty in cooling the liquors on mixing the aniline and chromic solutions. It is not desirable to use an aniline oil of higher specific gravity than 1·007.

The following industrial process is described by GIRARD and DE LAIRE:—100 kilos. of heavy aniline oils are mixed with 54 of sulphuric acid at 66° B., and 2 of water, the diluted sulphuric acid being added little by little, with continual stirring. When the whole is added, the mass is heated and stirred so as to obtain a homogeneous product, and then cooled: 140 kilos. of potassium dichromate dissolved in as little water as possible is then added to the cold liquid, and the whole allowed to stand twenty-four hours. At the end of this time a black precipitate is formed. The vats in which this operation is conducted are then filled up with water, so as to wash the precipitate by decantation; the washing is repeated three or four times, and the precipitate collected in filters and washed with water, acidulated with sulphuric acid, and finally with pure water; the residue is then boiled with water for three hours with continual agitation. A violet solution is thus obtained, which is filtered and precipitated with an alkali; the precipitate is washed with lukewarm water and dissolved in acetic acid. To obtain a pure product the process should be repeated several times. From 68 to 90 kilos. of thick mauve-paste may thus be obtained from 100 of heavy aniline oil (GIRARD and DE LAIRE); an average of 70 parts of paste, containing 10 per cent. of dry colouring matter (SCHLUMBERGER).

TABOURIN and FRANC use hydrochloride of aniline in lieu of sulphate in the preparation of PERKIN'S mauve.

GLAVEL prepares a soluble mauve by treating the colouring matter with fuming sulphuric acid, with continual agitation: after standing for half an hour the whole is greatly diluted with warm water and boiled. After cooling, common salt is added to precipitate the colouring matter, which is then collected and washed with water till the acid and salt are removed. It is wholly soluble in warm water, and the aqueous solution can be used direct for dyeing.

DALE and CARO'S process is worked as follows:—One equivalent of aniline hydrochloride, sulphate, acetate, or nitrate, and 6 of chloride of copper, are dissolved in water (30 times the weight of the aniline salt), and boiled till nothing more precipitates. This requires three to four hours. The black precipitate is then washed on a filter with a weak solution of soda until all chlorine is removed, and boiled with water as above. The filtered liquid is precipitated with a little soda, and the precipitate washed, when it is fit for use. Some dyestuff is left in the residue insoluble in water, whence it can be extracted by alcohol. A mixture of sulphate of copper and chloride of sodium, in equivalent quantities, answers as well as copper chloride.

Bleaching Powder Process.—This process, originally proposed by BOLLEY, and subsequently patented with slight variations by BEALE and KIRKHAM, may be worked as follows:—One part of aniline is dissolved in 1 of hydrochloric acid and 3 of water, and then added to 60 parts of a solution of bleaching powder of such a strength as to contain 1½ per cent. of its bulk of free chlorine gas; a black precipitate is thrown down, which is well washed, and dissolved in a little sulphuric acid. On addition to the latter solution of a large bulk of water, a precipitate is thrown down which is washed and purified as in PERKIN'S process. The product has frequently a redder tinge than the mauveine made by the bichromate process; but the yield is usually somewhat greater.

Ethyl Mauveine.—The hydriodide of ethyl mauveine has been sold as a dyestuff. Equal weights of mauveine and ethyl iodide are heated together to the boiling point for four to five hours; the excess of iodide is then distilled off, and the residue dissolved in 16 to 20 parts of boiling alcohol; after standing twenty-four hours, the liquor is filtered. It is noteworthy that this substance has a *redder* shade than mauveine, whilst ethylated rosanilines are *bluer* than rosaniline itself.

Manufacture of Violets de Lyons. — (Mono- and diphenyl- rosaniline). These violets are now almost wholly superseded by the HOFMANN and methyl violets. They were prepared some years ago by essentially the same process as that used for aniline blue, except that a smaller proportion of aniline was used and the operation was shortened. According to E. KOPP many of the violets in the market about 1865 were simply mixtures of substances containing unaltered fuchsine and aniline blue: such mixtures do not answer well for dyeing purposes (*Bull. Soc. Chim.* Paris, [2] iii. 153). GIRARD and DE LAIRE'S process was as follows: — Equal weights of purified aniline oil and of a rosaniline salt (arseniate, hydrochloride, oxalate, or acetate) were heated in a cohobator placed in an oil or paraffin bath for several hours to a temperature between 155° and 180° C. The violaceous mass thus produced was digested with warm dilute hydrochloric acid, which dissolved out the unaltered aniline and fuchsine, leaving behind a violet mass soluble in alcohol, wood spirit, strong acetic acid, or boiling dilute acetic acid. Any of these solutions can be used for dyeing.

According to the duration of the operation a red or a blue violet could be produced; these were sold as "Imperial red violet" and "Imperial blue violet" respectively. In permanency they are inferior to PERKIN'S mauve, but superior to the HOFMANN violet.

GIRARD and DE LAIRE have since described the following process for preparing these two dyes (*Traité des Derivés de la Houille*, Paris, 1873):—

*Red Violet or Dahlia.**—Fourteen kilos. of aniline free from moisture, and 10 of anhydrous sulphate or hydrochloride of rosaniline, are heated gently in the cohobator; for the first half hour the aniline is allowed to drop back, but afterwards the cohobator is shut off. The temperature is then raised, but is not allowed to pass 190° C.; samples are drawn every five minutes by means of a rod; when a tint is attained rather redder than that ultimately desired (as the purification operations remove unaltered rosaniline), the heat is withdrawn and the mass cooled. This takes from one and a quarter to one and a half hour's cohobation. While still fluid it is gently poured into excess of benzol with continual agitation. Unaltered aniline and brown and chestnut products remain dissolved, whilst rosaniline and the violet precipitate. The precipitate is pressed, dried, and dissolved in strong hydrochloric acid; water is then added, when the violet is precipitated and washed with pure water and dried, whilst the rosaniline remains in solution. The benzol containing aniline is treated with hydrochloric acid to throw down aniline hydrochloride and colouring matters, and is then distilled over an alkali.

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* This term has been applied to many kinds of dyestuff having approximately the same tint; notably to ethylated mauveine and to a product manufactured by a process kept secret, but stated to be obtained from residues in the fuchsine manufacture.

Instead of running the mass into benzol, it may be slightly diluted with alcohol and run slowly into acidulated brine; an impalpable powder then precipitates, which is well washed with fresh acidulated brine, and finally with pure water, after which it is pressed and dried at a temperature not above 40° C.

Blue Violet.—The operation is conducted just as for violet, using, however, a little more aniline (twice the weight of rosaniline salt, *i.e.*, 20 kilos. aniline and 10 rosaniline salt), and allowing a somewhat longer time, viz., from twenty to thirty minutes, so that the operation lasts one and a half to two hours. Too high a temperature must be carefully guarded against during the last quarter of an hour, and the sample must be drawn at very short intervals. When finished the mass is slightly cooled, and 4 to 5 litres of alcohol gently run in with continual stirring. The whole is then poured into 400 litres of alcohol acidulated with hydrochloric acid; after which brine is added to throw down the colour. Rosaniline and some violet red remain dissolved. The precipitate is washed first with dilute acid, and finally with pure water, till no longer acid. If the heating operations have been pushed too far the product is slightly resinous, and should be washed with weak alcohol; as also if the shade is a little too red. If much insoluble matter is present, the whole should be dissolved in strong alcohol in an agitating apparatus.

Manufacture of Hofmann's Violets. — The three varieties, marked R, B, and B B, of these violets, are prepared in much the same way, the chief difference in the processes being the proportion of the alcoholic iodide used. In the early days of the manufacture the process was conducted under the ordinary atmospheric pressure, in a digester provided with a cohobater and condensing worm and an agitator, and surrounded by a steam jacket. At the present day autoclaves are usually employed, and the action is brought about under a greatly increased pressure. After about two hours' heating at about 115° to 130° C., the operation is finished. The alcohol and excess of iodide is distilled off, and the residue is ready for the market if the hydrodides of the resulting bases are required.

Instead of heating by steam a saline bath may be used. Thus, a solution of calcium chloride may be used, or even brine. If the temperature be lower than the above, a longer time is requisite; thus, four or six hours, or even more, at 100° to 110° C., is required. When the digester is opened permanent gases (ethylene and methylic ether?) escape; the manhole must therefore be opened cautiously, otherwise effervescence and loss of material ensue. For the three qualities the following proportions may be used (Girard and De Laire):—

	R	B	BB
Rosaniline, . . .	10 kilos.	10 kilos.	10 kilos.
Alcohol, ...	100 litres.	100 litres.	100 litres.
Methyl or ethyl iodide,	8 kilos.	5 kilos.	20 kilos.
Caustic potash or soda,	10 "	10 kilos.	10 "

According to the patent specification of Hofmann (May 22, 1863), the following proportions may be used for violet:—1 part of magenta, 2 parts of ethyl iodide, and 2 of methylic or ethylic alcohol.

Ethyl iodide does not give the bluer shades so readily as methyl iodide; hence, for the B B colour methyl iodide only is usually employed. If, however, methylic alcohol be employed together with methyl iodide, methyl iodide and ethylic alcohol are formed; thus—

$$CH_3.OH + C_2H_5I = C_2H_5.OH + CH_3I.$$

So that blue shades may be obtained with methyl iodide if wood spirit, or even ordinary methylated spirit, be employed along with it.

If the hydriodides of the violet bases are used in dyeing, it is necessary to employ alcohol as a solvent, the salts being insoluble in water. In addition, the high price of iodine at the present day renders it necessary to recover the iodine; *i.e.*, to convert the hydriodides into other salts. For this purpose the crude hydriodides are boiled with alcoholic caustic soda in a digester; the mass is then washed with hot water to separate sodium iodide and excess of caustic soda, and dissolved in hydrochloric, sulphuric, or acetic acid. From this filtered solution the dyestuff is thrown down by common salt, and is collected and dried. The product is perfectly soluble in cold water if pure.

Some manufacturers employ the alcoholic bromides in lieu of the iodides. The hydrobromide and ethyl bromide of triethyl rosaniline are sometimes met with in commerce.

As already stated, the violet B B consists chiefly of a salt of trimethyl rosaniline. In addition the salts of the mono- and tri- methylates of trimethyl rosaniline are usually present also to a small extent. Both of these are violet blue dyes.

Some few dyes have likewise been prepared by processes similar to those used for making Hofmann's violets. Methylated and ethylated mauvanilines are thus prepared from mauvaniline; benzyl chloride violets from rosaniline and benzyl chloride; benzyl-methyl violet from Hofmann's violet R and benzyl chloride; phenyl-methyl violet from violet de Lyons and methyl iodide; and isopropyl, amyl, allyl, and other analogous violets from isopropyl, amyl, allyl, &c., iodides, and rosaniline.

Turpentine Violet (*Britannia Violet*).—The reaction employed by Perkin in the production of this violet has been used in the preparation of other analogous products. Turpentine is dropped into bromine water, and the whole shaken together till no free bromine is left. One part of the brominated turpentine, 1 of rosaniline, and 6 of wood spirit or alcohol, are then heated in an enamelled autoclave for eight hours to 140° to 150° C. After cooling, the product is dissolved in alcohol or other like solvent. A red shade is obtained with 2 parts brominated turpentine, 3 of rosaniline, and 15 of alcohol; a blue shade, with a larger proportion of brominated turpentine.

Levinstein's Dorothea is formed by heating together at 100° C. for two to three hours a mixture of 90 per cent. alcohol, 50 parts; rosaniline, 35 parts; and nitric ether, 9 parts.

Manufacture of Methyl Violets.—The following process is described in the patent specification of POIRRIER and CHAPPAT:—Mix the methyl and dimethyl aniline obtained by their process with five to six times its weight of chloride of tin perfectly free from water, and heat to 100° C. for several hours, when the mixture becomes solid and hard. The product is then cooled and boiled with caustic soda ley. The free base remains undissolved, and can be obtained as salt by solution in any acid. Mercuric chloride can also be used. The exact point at which the heating must be stopped is known by practice. If the heating be carried on too long, much black tarry bye-products are also formed.

The following processes are also described in a later specification:—A mixture of 100 parts methyl aniline sulphate, 100 to 150 of powdered potassium chlorate, and 100 to 150 of water, is heated for several hours at 160° C. Chloride of iodine may be used in place of chloride of tin; or a mixture capable of producing chloride of iodine, such as methyl aniline, 100 parts; iodine, 20 parts; and potassium chlorate, 20 parts.

The following mixtures may also be used:—Methylaniline, 1 part; mercuric chloride, 1½ part; and potassium chlorate, 1 part; or, methyl aniline, 1 part; mercuric iodide, 3 parts; and potassium chlorate, 1 part; or, methyl aniline, 1 part, and benzene hexachloride $(C_6H_6Cl_6)$, 3 to 4 parts; in this last case the heating should be effected at 150° to 160° C. Blue and green tints can be produced by further treatment of the products with methylic, ethylic, &c., iodides. (*Bull. Soc. Chim. Paris* vi. 502.)

LAUTH has patented the following processes:—Methyl aniline is heated to 120° C. with half its weight of hydrochloric acid for as long a time as any colour is formed, or to 100° C. with half its weight of nitrate of copper; or 2 parts of methyl aniline acetate is heated to 100° C. with 1 part of mercury oxide. The longer the heating is continued, the bluer the shade. He also states that methyl violet is readily producible by simply heating a mixture of a methyl aniline salt and sand to 100° to 120° C. Ten parts of methyl aniline, 3 of hydrochloric acid, and 200 of sand answer well. The mass simply requires to be cooled with water, which extracts the colouring matter. Care must be taken to employ in the preparation of the methyl aniline used an aniline oil boiling almost wholly near 182° C., as oil containing more than 5 per cent. of higher homologues of aniline is not suitable for the production of good colours.

HOFMANN uses a mixture of 10 parts of dimethyl aniline, one of potassium chlorate, 2 of copper sulphate, and 100 of fine sand, and heats on the water bath for several days. Water and aniline are evolved. The following mixture may likewise be used:—dimethyl aniline, 10 parts; common salt, 2 parts; copper nitrate, 1 part; water, 1 part; and fine sand, 100 parts. These are well mixed. After an hour 2 parts of glacial acetic acid are added, and after two or three hours the mass is made into cakes and dried at a temperature not exceeding 50° C. for two days. The mass is then digested with a solution of sulphide of sodium of sp. gr. 1·162, 1 part of liver of sulphur being used per 10 parts of dried cake. The residue is well washed with cold water, dissolved in a large bulk of boiling water, and the dyestuff precipitated from the liquor by common salt.

Preparation of Regina Purple (*Nicholson's Purple*).—NICHOLSON describes the following process in his patent specification, dated 1862:—Aniline red is heated to between 390° to 420° Fahr. (199° and 215° C.) in a suitable apparatus. The substance quickly assumes the appearance of a dark, semi-solid mass, the red dye being transformed into a dark substance with evolution of ammonia. The mass is extracted, preferably with acetic acid about equal in quantity to the red dye used, and the acid solutions are then diluted with sufficient alcohol to make a dye of convenient commercial strength. The violet liquor may be used directly for dyeing.

Emeraldine. [*Vide* AZURINE.] This term has also been applied to the following dyestuff.

Manufacture of Aldehyde Green.—One kilo. of rosaniline is dissolved in 2 of sulphuric acid of specific gravity 1·63, previously diluted with 500 grammes of water: 4 kilos. of a concentrated alcoholic solution of aldehyde are then added in small quantities at a time. In from fifteen to twenty minutes the reaction is finished and the blue colour developed; the mixture is next thrown into 85 litres of water containing in solution 4 kilos. of sodium thiosulphate (hyposulphite), and the whole boiled for seven or eight minutes and filtered. A bluish grey substance is retained, whilst a solution of the green passes through; the filtrate is then precipitated by either sodium acetate, tannin, oxide of zinc, or a zinc salt and an alkali; the precipitate is washed with water, dissolved in alcohol, and thence precipitated by an alkali.

The bluish grey insoluble substance frequently forms the larger part of this product; it is often sold under the name of *Argentine.*

Many other processes, slightly varying from the above, have been given. Thus, BOLLEY gives the following formula for the preparation of the aldehyde blue in the first instance: 20 parts of crystallized fuchsine are dissolved in 280 of commercial hydrochloric acid; an equal bulk of water is added, and 100 parts of crude aldehyde, and the whole allowed to stand for twenty-four hours.

USÈBE gives another recipe, viz.: rosaniline sulphate, 150 grammes; strong sulphuric acid, 3 kilos.; water, 1 kilo.; crude aldehyde, 225 grammes. Dissolve the rosaniline salt in the acid and water, gradually mix the aldehyde, and then add the liquor to a solution of 450 grammes of sodium thiosulphate in 30 litres of hot water, and boil for a few minutes. Another mixture is: rosaniline salt, 300 parts; diluted sulphuric acid (3 parts acid, 1 water), 900 parts; aldehyde, 450 parts; sodium thiosulphate, 900 parts; dissolved in 60,000 parts of water.

Lucius takes 1 part of rosaniline sulphate, 2 parts of sulphuric acid, 2 to 4 of water, and 4 of aldehyde. This mixture is allowed to stand at 50° C. until a sample dissolves in 50 times its weight of alcohol with a green-blue colour: from 300 to 500 parts of a saturated aqueous solution of sulphuretted hydrogen are then added to the whole, which is slowly heated to 90° to 100° C.: 10 parts of saturated aqueous solution of sulphurous acid are then added, and the liquor filtered to separate a blue precipitate.

Hirzel employs ammonium sulphide in lieu of thiosulphate.

Aldehyde green is usually prepared by the dyer himself as required; as the dye-stuff will not keep well, becoming dull in colour and finally useless, it is rarely manufactured for the market.

Schlumberger prepared a very similar product, *Toluidine green*, by treating Coupier's rosatoluidine by the same process as that employed in the preparation of aldehyde green from fuchsine.

Manufacture of Iodine Green.—To produce this dyestuff, Keiffer dissolves 1 part of Hofmann's violet in 3 of alcohol, adds from ½ to 1 part of ethyl iodide, and then heats in a digester or in a cohobating vessel for half an hour; aqueous caustic soda or potash is then added, and the whole boiled for three to four hours. The residue is washed (the wash-waters being retained for the recovery of the iodine), and boiled with 500 to 600 parts of water; the filtered fluid is then precipitated with picric acid.

Wanklyn and Paraf heat equal quantities of rosaniline, wood spirit, and ethyl iodide (or isopropyl iodide or other homologues of ethyl iodide) to 110° to 115° C. for from three to four hours in an autoclave or cohobator, and then boil with 4 to 5 times their weight of water containing 1 per cent. of soda. Some green is thus dissolved, whilst much Hofmann's violet remains; this is again treated with equal quantities of wood spirit and ethyl iodide, and so on as before. Most of the violet is thus transformed into green, which is dissolved by the soda ley.

To obtain a good product it is essential that water should be wholly absent from the materials in the first part of the process, that no free alkali should be present, and the temperature should not rise above 100° C., all of which conditions are violated in the method adopted for Hofmann's violet manufacture; hence but small quantities of this substance are formed as bye-products during the preparation of these dyes, although it invariably accompanies the bluer shades. Ethyl iodide does not give a good yield, nor is the product of good quality. The nature of the rosaniline salt has some influence on the reaction, the acetate being the most suitable, and after that the sulphate, nitrate, and benzoate.

The manufacture from methyl iodide is thus described by Girard and De Laire (*Traité des Dérivés de la Houille*, Paris, 1873):—10 kilos. of pure rosaniline acetate, 20 of methyl iodide, and 20 of methylic alcohol boiling at 64° to 70°, are introduced into an enamelled iron autoclave, capable of bearing a pressure of from 20 to 25 atmospheres. Steam is passed through the worm in the water jacket, the pressure never being above 1 atmosphere, and hence the water not being heated beyond 100°. If the temperature rise above 100° the methyl green is decomposed into mono-methyl iodide of trimethyl rosaniline, and free iodide of methyl. As the reaction goes on, a considerable pressure is developed in the autoclave, rising quickly to 8 atmospheres, and then slowly to 10 or 11, beyond which it should not go. After between four and five hours the steam is cut off, and the whole cooled down; in twelve hours more the internal pressure is not above 4 atmospheres; a condensing worm is then attached, and the screw-cock slightly opened. Methyl iodide and acetate, and much methylic ether, with the excess of methylic alcohol, distil over; the semi-fluid product is transferred to a jacketed vessel heated by steam, and dissolved in 600 litres of water.

The green dimethyl iodide of trimethyl rosaniline thus passes into solution, whilst most of the violet derivatives are left undissolved. The liquid is filtered, 24 to 25 kilos. of common salt added, and crystallized sodium carbonate added in small quantities to the boiling liquor; the small quantity of violet products retained in solution by the acid set free during the reaction, is precipitated as the acid becomes neutralized. When a sample of the liquid filtered through sand shows a pure green tint free from all trace of blue or violet, a cold saturated solution of picric acid free from nitric acid, containing 3·46 kilos. is added. A picric acid compound of the green dye is thus thrown down, sparingly soluble in water; this is collected, slightly washed, and sold as a paste. Being so sparingly soluble, alcohol must be used as a solvent; moreover, the picric acid gives a strong yellow cast, hence it is preferable to use other precipitants. Zinc chloride, sulphate, or acetate, form more soluble double compounds, and are hence much used for this purpose. They are usually sent into the market dry.

A crystallized green is sometimes prepared by dissolving the crude product in much less boiling water than that above stated, and allowing it to cool after more completely saturating with carbonate of soda. Silky needles are then deposited on copper rods placed in the liquor, a less pure crystalline magma being found at the bottom of the vessel. These crystals are drained, slightly washed with water to remove adhering brine, and dried at the ordinary temperature.

Of late, iodine green has been sold as a chlorinated compound, prepared by setting free the dimethyl hydrate from the dimethyl iodide by an alkali, and then dissolving in hydrochloric acid.

Sixty per cent. of the total yield is obtained by the above process as green dye, and forty as a violet dye, chiefly mono-methyl iodide. This may be purified and sold as a violet, but is usually transformed into green. A finer and purer green dye is thus obtained than by the direct treatment of rosaniline. The violet mass is well washed, dried, and pulverized, dissolved in alcohol, and transformed into free base by alcoholic caustic soda. It is then heated with the equivalent amount of methyl iodide to a temperature not above the boiling point of the latter, namely, 48° C. Combination takes place quickly; and when it is completed (known by the tint of a sample drawn), the product is cooled and acetic acid added. The whole is then thrown into a large bulk of warm water. A little unaltered violet is thus precipitated. The solution is purified as before.

Methyl Green.—Poirrier and Chappat describe the following process (Patent No. 72561: *Bull. Soc. Chim. Paris*, [2] vi. 504).—Heat .in a close vessel, for about twenty-four hours, to 100° C., a mixture of 1 part Paris violet, 2 parts of alcoholic iodide, 2 of methylene (methylic alcohol ?), and $\frac{1}{5}$th of sulphuric acid. The mass is next saturated with an alkali, when the green colour is precipitated; this is separated by filtration, whilst a blue is retained in solution. Bromides may be used instead of iodides. As regards this latter point, Hofmann and Girard have shown that bromide of methyl or ethyl may be readily formed by the action of methylic or ethylic alcohol on amyl bromide, amylic alcohol and methyl or ethyl bromide being produced thus—

$$C_5H_{11}Br + CH_3,OH, = C_5H_{11},OH + CH_3Br.$$

By using a mixture of methylic alcohol and amyl bromide, instead of methylic iodide, the same result is arrived at by cheaper means, the price of bromine being far less than that of iodine, and the practical inconveniences attending the greater volatility of the alcohol radical bromides being thus obviated.

"Ethyl green" from Paris violet is frequently of a much finer shade than ordinary iodine green. It is also stated that methyl green is more stable in daylight than iodine green. Whether this arises solely from greater purity, or points to the substances not being actually identical, but only isomeric, is not at present known.

Methyl chloride, or a mixture of materials that will give rise thereto, such as a mixture of methylic alcohol and sal ammoniac, may be substituted for methyl iodide. In this case the corresponding trimethyl rosaniline dimethyl chloride is formed; this gives a well-crystallized zinc salt.

Manufacture of Various Browns.—Girard and De Laire's Brown.—Four parts of anhydrous aniline hydrochloride are fused, and one of dry fuchsine or aniline red is gradually added. When completely dissolved, the temperature of the mixture is raised to the boiling point of aniline hydrochloride, namely, about 240° C. The mass is allowed to boil until the colour (which is at first a violet red) suddenly changes to chestnut. The operation lasts from one to two hours, no alteration apparently taking place at first; at the close a yellowish vapour is emitted, and at the same time a peculiar odour is perceptible, resembling that of phenyl cyanide.

The resulting chestnut is soluble in water, alcohol, and acids. It may be either used immediately (*Direct brown*), or may be dissolved in water, and the dyestuff salted out with common salt, or almost any other saline solution, or a free alkali. It gives fine shades, especially on leather and silk.

Girard and De Laire consider that this substance is probably a chrysotoluidine derivative, formed from the original fuchsine by the action of toluidine on the aniline salt used.

Wise's Brown.—A mixture of equal weights of formic acid and fuchsine is heated to 140 to 210° C. for several hours; an orange red mass results. This is allowed to cool, and then again heated with 3 parts of aniline to about 230° C. for an hour or two. The mass is purified by dissolving it in dilute hydrochloric acid, and salting out the colouring matter in the usual manner.

Another process is to employ rosaniline, 2 parts; formic acid, 2 parts; and sodium acetate, 1 part: heat as before, again treating the product with 3 parts of aniline.

Jacobson obtains an analogous brown by heating to 100° C. a strong solution of ammonia chromate, mixed with aniline formate; or by heating to 140° C. 1 part of picric acid and 2 of aniline, as long as ammoniacal vapours are evolved. The product is dissolved in hydrochloric acid, and precipitated by soda.

Sieberg's Brown.—The impure fuchsine obtained from rosaniline mother liquors (sometimes known as *Cerise*) is added to twice its weight of melted aniline hydrochloride, and the whole heated on a sand bath till the brown tint appears. The product is well stirred with 4 parts of soda crystals dissolved in 25 times as much water, and the undissolved portion well washed with water. When used, 1 part of dry colour is dissolved in 9 of alcohol, 13 of water being then added.

Probably all these browns are more or less impure chrysotoluidine, or its substitution derivatives. Knap brought into the market in 1869 two dyestuffs of this description, one chestnut, under the name of *Marron*; the other approaching to orange-yellow, termed *Vesuvine*. *Cannelle Brown*, *Manchester Brown*, *Bismarck Brown*, and numerous other names, have been given by various manufacturers to similar products.

Leucaniline Browns.—Durand prepares leucaniline from fuchsine by boiling with zinc dust; the insoluble mass thus formed is boiled with alcohol, and the alcoholic extract evaporated to dryness, when a resinous yellow mass is left. Köchlin mixes this with sulphide of copper, and prints on cotton in just the same way as in the preparation of aniline black. Fayolle employs the mother liquor of fuchsine, and prepares an impure leucaniline salt by treatment with zinc and sulphuric acid. Caustic soda, or common salt, precipitates from the resulting liquor a mixture of products which dye yellow, nankeen, leather colour, and brown shades; the tints are darkened by passing the goods through a bath of potassium dichromate after dyeing.

Preparation of Aniline Grey.—Castelaz prepares this dye by dissolving 10 kilos. of mauveine in 11 of strong sulphuric acid (at 66° B.); and when complete solution has been effected, adding 6 kilos. of aldehyde.

The whole is left for four to five hours, and is then poured into water, in which the grey colouring matter dissolves. The dyestuff is then salted out, and the precipitate purified by solution in water, and salting out several times successively. This dyestuff yields good tints, but is costly. Its composition is unknown. It is stated to require no mordant even with vegetable fibres. Faint shades of pearl grey may be communicated to silk and wool by means of very weak solutions of aniline violets; the finer the violet, the purer the grey.

Block prepares a grey by heating together 1 quart of aniline and 5 of fluid arsenic acid until foaming begins. The crude product is treated with a boiling mixture of 20 parts water and 5 hydrochloric acid. The undissolved portion is washed first with water, and next with dilute soda liquor, and then dissolved in alcohol containing 10 per cent. of sulphuric acid. Wool and silk are dyed with a very minute quantity of the product. (*Jahresbericht*, 1869, 1165.)

Carvès and Thirault mix a solution of 1 part of aniline in 2 of hydrochloric acid, with a mixture of ½ part bichromate of potassium, and ½ part of ferrous sulphate, dissolved in 1 part sulphuric acid and 3 of water. A viscid mass separates; this is washed with cold water and dissolved in boiling water. The solution dyes greys which withstand soap and acids. (*Jahresbericht*, 1867, 964.)

ANTIMONY.—*Antimoine*, *antimonium*, French; *antimon*, *spiessglanzmetall*, *spiessglassmetall*, German; *stibium*, Latin. Symbol, Sb.; atomic weight, Rose, 120·7; Dexter, 122·3; Schneider, 120·3.—The stibium of the Romans was the sulphide of this metal. It was used for painting the eyebrows, and Eastern ladies still apply it for the purpose of imparting by contrast a fictitious freshness to the complexion.

The principal ores of antimony are found in the mines of Bohemia and Hungary, in France, England, and in America. During the age of alchemy various other antimonial compounds were discovered; and an account of a process of preparing the metal was given by Basil Valentine in 1460.

Metallic antimony is met with abundantly in nature—native, combined with other metals: namely, with a little silver and iron in native antimony; with silver in antimonial silver; and with arsenic in arsenical antimony.

Antimony is also largely found combined with sulphur and the sulphides of other metals; for example, *per se*, in grey antimony, which generally contains copper, arsenic, and iron; with iron in Berthierite; with nickel and arsenic in nickeliferous grey antimony; with lead and traces of iron, copper, bismuth, and zinc in Jamesonite; with lead, copper, and iron in Bournonite; with lead and a trace of copper in zinkenite; with lead in antimonial lead glance; with silver and copper in melanglanz; with silver, copper, and iron in miargyrite; with silver in dark ruby silver, rhombohedral ruby blende; with silver and arsenic in light ruby silver; and with arsenic, silver, and iron in arsenical silver. It is also found, more or less, in the following argentiferous minerals: namely, with silver, copper, iron, and zinc, in weissgültigerz and graugiltigerz; with copper and silver in antimonial grey copper, tetrahedral copper glance; and in very minute quantity, with copper, iron, silver, and arsenic, in kupfer-blende. It occurs as an oxide in white antimony, which is sometimes contaminated with sesquioxide of iron, and with sulphide of antimony in the red antimonial ore; also as antimonious acid in antimonial ochre.

Antimonial copper—*Antimonkupferglanz*, German—is composed, according to Schrötter's analysis, of—

	Centesimally.
Lead,	29·902
Copper,	17·312
Antimony,	16·644
Arsenic,	6·036
Iron,	1·404
Sulphur,	28·702
	100·000

This composition does not conveniently admit of being represented by a chemical formula.

The principal sources of the antimonial ores are the Continental mines, very little of the metal being found in this country. The ore submitted to reduction is the sulphide of antimony—grey antimony—of which from 600 to 1000 tons are annually imported into England.

In the laboratory the metal is prepared in a variety of ways; if 8 parts of powdered sulphide of antimony be intimately mixed with 6 parts of cream of tartar (potassium acid tartrate), and 3 of nitre, and the mixture be thrown in small portions into a red-hot crucible, and kept at redness for some time, a metallic button collects at the bottom. The antimony thus obtained is, however, impregnated with small traces of iron, and resembles the better sort of the crude metal found in the market.

When the sulphide of antimony, mixed with some charcoal to prevent the caking of the mass, is heated in contact with air, the greater part of the sulphur is expelled and antimonious oxide (Sb_2O_3) formed; this may now be reduced to metal, either by fusing with an adequate quantity of black flux, or with the addition of ordinary soap; a button of impure metal remains. The purest antimony is procured by reducing this oxide either with powdered charcoal and alkali, or the potassium acid tartrate at a proper degree of heat.

As met with in commerce, antimony is more or less impure, in consequence of some imperfections or mismanagement in the process of its reduction. The contaminations are generally lead, iron, arsenic, and sulphur; but these may be separated by grinding the crude metal in an iron mortar, and fusing the powdered mass with one-tenth of its weight of saltpetre, which oxidizes all the foreign ingredients, as well as a part of the antimony, leaving the remainder in a pure state.

Antimony is very brittle, possesses a white silvery aspect and a plated crystalline texture, so that, when broken up, the fracture exhibits beautiful facets. It happens, also, that when the metallic button of antimony is suffered to cool in the crucible, the surface presents a fine stellated appearance. In consequence of this crystalline property, the alchemists considered the phenomenon symbolical of that mysterious luminary which was to guide them in their occult path to the discovery of a universal medicine, or the philosopher's stone.

At 800° Fahr. the metal fuses, and at a white heat it slowly but distinctly volatilizes in closed vessels; in a current of hydrogen, however, it distils with tolerable facility. If exposed to a stream of oxygen on ignited charcoal it burns brilliantly, evolving a white light, and is at the same time converted into the tetroxide (Sb_2O_4), which is eliminated in the form of a thick whitish smoke. Should a globule of the metal at a high temperature be dropped from an eminence upon a board or plate of iron, the mass subdivides into numerous smaller globules, which hop along the plane in a combustible state, marking the course they take by a white streak or line of the tetroxide.

Antimony is not sensibly affected on exposure to the air at the ordinary temperature; its surface becomes slightly tarnished, but does not rust. If exposed to the air during fusion, rapid oxidation takes place.

Hydrochloric acid dissolves antimony with the evolution of hydrogen and the formation of the trichloride ($SbCl_3$): concentrated sulphuric acid oxidizes it, sulphurous acid being at the same time evolved; but if the acid be diluted with water it does not dissolve it. Nitric acid, even when very dilute, rapidly attacks antimony, and converts it into a white insoluble powder which is the trioxide, tetroxide (Sb_2O_4), or the pentoxide (Sb_2O_5), according to the strength of the acid and the time of digestion. Aqua regia converts the metal into the trichloride.

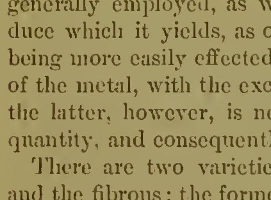
The specific gravity of antimony is 6·712, and its specific heat, according to REGNAULT, is 0·05077.

For preparing the metal on a large scale, the trisulphide (stibnite, or grey antimony ore, Sb_2S_3) is generally employed, as well for the abundant produce which it yields, as on account of its reduction being more easily effected than that of any other ore of the metal, with the exception of the native oxide; the latter, however, is never met with in sufficient quantity, and consequently is seldom used.

There are two varieties of stibnite, the massive and the fibrous: the former presents a long columnar composition, and the latter is distinguished by its plumous, woolly, or felt-like appearance; it is of a light lead colour, sometimes dull externally, and often iridescent.

The mineral is met with in crystals of the rhombic prism (Fig. 1), which are sometimes aggregated laterally. Before the blowpipe, on charcoal, sulphide of antimony fuses and is absorbed, giving off at the same time sulphurous acid and white fumes of the trioxide of the metal.

Fig. 1.



The most celebrated localities productive of this mineral are Felsobanya, Chemnitz, and Cremnitz, in Hungary, whence the grey antimony is procured; Dumfriesshire in Scotland, where the fibrous and laminated variety is found; and Cornwall, where the massive sort is raised. Other varieties of the sulphide are also to be found, at Magurka in Hungary, at Freyberg and Braunsdorf in Saxony, and at Stolberg in the Hartz.

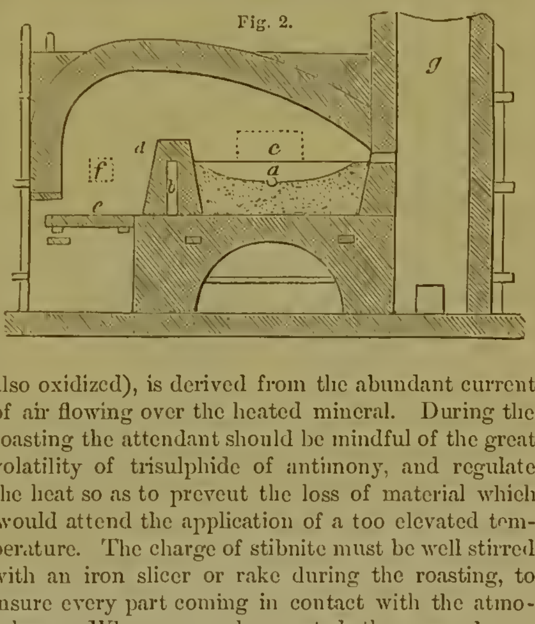
MANUFACTURE OF ANTIMONY ON THE LARGE SCALE.—Many of the operations in the preparation of antimony on the large scale are the same as those which will be mentioned hereafter, when describing the dry assay of the antimonious compounds. The method most commonly followed is, first to convert the sulphide into oxide, and then to reduce with cream of tartar.

The sulphide of antimony is deprived of its sulphur, and oxygen substituted by heating with free access of air, as in the annexed equation:—

$$\overset{\text{Antimonious sulphide.}}{Sb_2S_3} + \overset{\text{Oxygen.}}{9O} = \overset{\text{Antimonious oxide.}}{Sb_2O_3} + \overset{\text{Sulphurous oxide.}}{3SO_2}$$

This change is conducted in a reverberatory furnace, where the comparatively large amount of oxygen required to displace the sulphur (which is also oxidized), is derived from the abundant current of air flowing over the heated mineral. During the roasting the attendant should be mindful of the great volatility of trisulphide of antimony, and regulate the heat so as to prevent the loss of material which would attend the application of a too elevated temperature. The charge of stibnite must be well stirred with an iron slicer or rake during the roasting, to insure every part coming in contact with the atmosphere. When properly roasted the mass has a greyish-white appearance.

Fig. 2.



Having obtained the oxide in the manner described, it is intimately mixed with about a tenth of its weight of cream of tartar, and reduced in large earthen crucibles, heated in a wind furnace, when the following decomposition takes place:—

$$\overset{\text{Antimonious oxide.}}{Sb_2O_3} + \overset{\text{Potassium acid tartrate.}}{2KC_4H_5O_6} = \overset{\text{Antimony.}}{2Sb} + \overset{\text{Potassium carbonate.}}{K_2CO_3} + \overset{\text{Carbonic oxide.}}{7CO} + \overset{\text{Water.}}{5H_2O}$$

The antimony thus obtained is tolerably pure, and is ready for market. Iron is the principal, and often the only impurity present.

At Linz, in Germany, a furnace, similar to that represented in the annexed cuts, was constructed for the reduction of antimony at one operation. Fig. 2

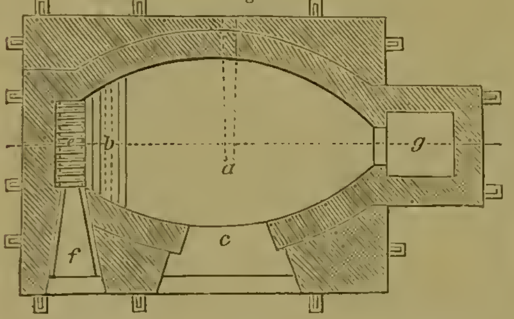
shows a vertical section; the bed is concave, and is constructed of sand and clay well consolidated; at its middle a pipe, *a*, forms a canal for the fused metal to flow out as soon as produced, and which is closed by heavy ashes. *b* is the air channel in the bridge *d*, and *c* is a door through which the prepared ore is introduced and the residuary slag abstracted. The fire is shown at *f*; *e* is the grate and *g* the chimney.

Fig. 3 is a plan of the furnace, wherein the same letters indicate the same parts.

Sulphide of antimony is dressed for reduction by mixing it with the proper quantity of iron, or iron scales, and alkaline carbonate: 2 or 3 cwts. of ore require from eight to ten hours' heat for reduction; but the metal obtained in this way is very impure, and is therefore remelted in portions of from 20 to 30 lbs. in crucibles. These are covered over with ground charcoal, and heated in a reverberatory furnace.

The metal obtained by this process contains considerable quantities of iron, sulphur, and frequently arsenic, lead, and copper. These bodies deteriorate its quality, even for the arts, and of course it cannot be employed in medicine while the smallest trace of arsenic is present. It is therefore imperatively necessary to subject the crude antimony to purification. For this purpose it is fused with $\frac{1}{8}$ part of antimonious sulphide, and an equal weight of sodium carbonate, in a hessian crucible for one hour. On breaking the crucible after cooling, the metal is carefully separated from the slag, and submitted to a second fusion with $1\frac{1}{2}$ part of the carbonate. Sometimes a third fusion is required; but in this case it is to be performed with 1 part of the alkaline carbonate. Should there be lead, it remains in the antimony after the second fusion, although the other metals may be completely removed; and from its great fusibility, slight portions are retained even after the third operation.

Fig. 3.



If arsenic be absent, a part of the metallic antimony is oxidized and remains in the slag; but very little, if any, of the metal is thus acted upon whilst arsenic is present.

It has been found that 100 parts of the crude metal and 6 parts of the sulphide yield 94 of pure antimony. It is necessary, when larger quantities of iron are present, to add proportionally more of the sulphide of antimony; for unless there be sufficient sulphur to combine with the whole of the iron, the arsenic cannot be oxidized.

Wöhler recommends deflagration of 10 parts of the metal with 12 of saltpetre, and 10 of carbonate of soda; the arsenic is oxidized, and forms potassium arsenite, and the antimony is at the same time converted into potassium antimoniate by this treatment. On boiling the fused mass with water, the arsenical compound is dissolved, leaving the antimoniate, which is collected, washed, dried, and reduced in the usual way with charcoal in an earthen pot.

The metal obtained by this means still contains copper and iron.

According to Berzelius, the impurities are removed by fusing 2 parts of the metal (finely powdered) with 1 part of the trioxide of antimony; the iron, copper, and arsenic are oxidized, and remain in the slag; in this case the lead, should any be present, is still combined with the antimony. Muspratt thinks it better to fuse rather more than 4 parts of antimony with 1 part of manganese dioxide, and to subject the regulus so obtained to a second fusion with one-tenth of its weight of sodium carbonate.

As the stibnite is nearly always found associated with seams of silicious and calcareous impurities, a preliminary operation is necessary in order to separate it from its gangue; this process is called *eliquation*.

Fig. 4.

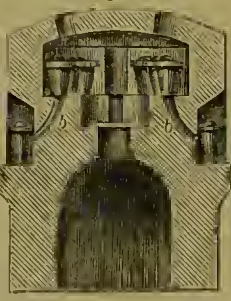
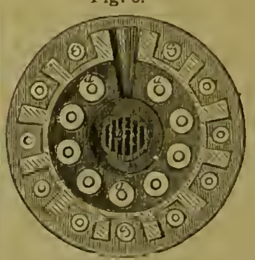


Fig. 5.

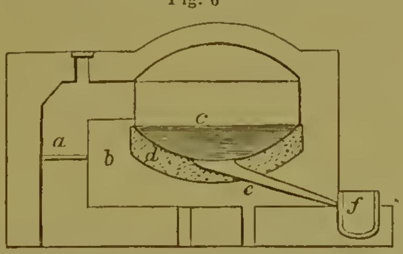


Any difference that exists in the manner of eliquating crude sulphide of antimony is chiefly in the furnace employed. In Hungary the ancient method, viz. that of roasting the ore in large pots or crucibles, is still practised. These pots are furnished with an aperture at their bottom, opening into a smaller one, which is embedded in the ground. A cover is appended to the upper pot, which may be luted down after the vessel is charged. These pots are placed in a circle with a furnace in the centre, the heat from which passes freely under the bottom and round the body of each pot.

Figs. 4 and 5 are drawings of this arrangement; the former is a section of the elevation, and the latter a plan, showing the position of the crucibles round the fire. *a a a* denote the pots in which the crude ore is heated, and *c c c* the smaller pots which receive the liquid sulphide; *d* is the fire-grate. In the elevated section, the relative position of these crucibles is comprehensively shown, with their connecting tubes, *b b*; both crucibles and tubes are made of earthenware.

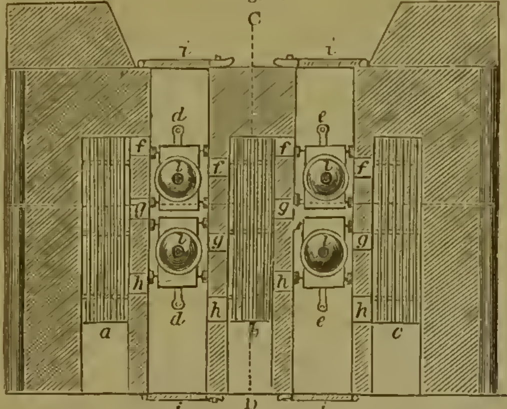
The furnace employed for the fluxier of the crude sulphide at Vendée, in France, is represented in Fig. 6, where, instead of several pots being placed round the fire, the bed of the reverberatory, which is connected with a receiver for the purified trisulphide, supplies their place. In the figure, *a* represents the fire-place, *b* the bridge of the furnace, *c* the concave hearth, lying on the substrata, *d*; *e* is the pipe which issues from the bottom of the furnace hearth, and carries the liquid products along to the recipient, *f*. The furnace is heated by the flue passing around the body, and the charge is introduced through an opening in the dome, not shown in the woodcut. This construction of furnace is far preferable to the preceding one, both for the simplicity of its arrangement and the advantage of its being reverberatory. When moderately rich ores are subjected to purification, the charge, which is usually from 8 to 10 cwts., yields from 4 to 5 cwts. of the purified trisulphide, with a consumption of about 25 cubic feet of wood as fuel. Three such charges may be worked off in the long days of summer.

Fig. 6



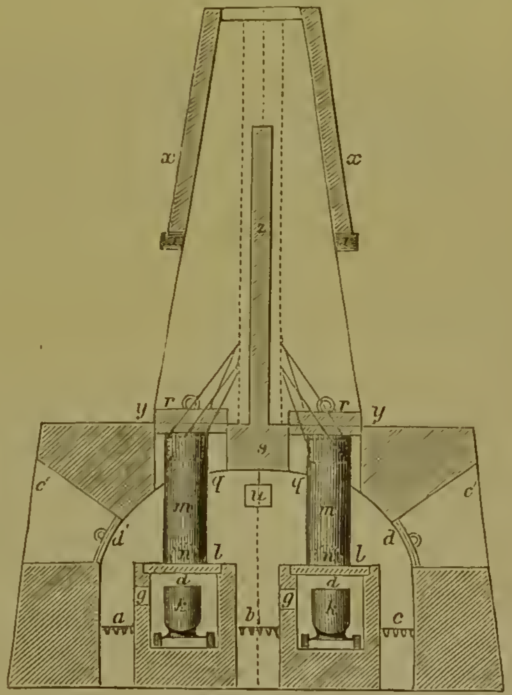
The best furnace for the separation of the sulphide of antimony from its matrix, is that in use at the extensive mines of Malbose, in the Ardèche Department in France; it is sketched in the subjoined engravings. Fig. 7 is the plan of the furnace on a line with the flues *f g h*, which issue from the several fires, and the grates *a b c*, whereon the ignited fuel is supported. These grates are 4½ feet in length by about 1 foot in breadth, and are separated by two square galleries, *d* and *e*, running parallel through the furnace, and protected from the direct action of the fire by walls, having the alternate openings, *f g h*, serving as flues, to conduct the heat into the galleries, which are closed by the iron doors, *i i*, at each end. Fig. 8, the section of the furnace explains the nature of the arrangement.

Fig. 7.



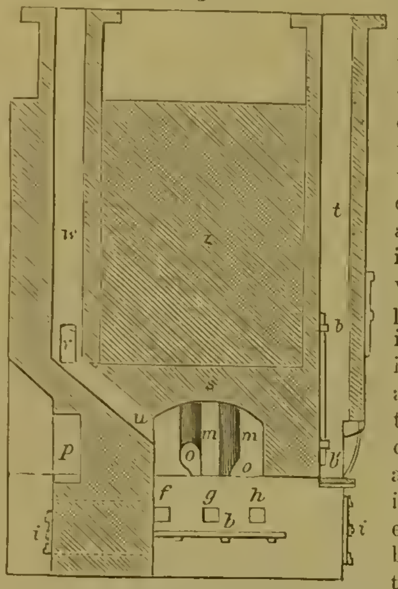
In this drawing all the parts before referred to are indicated by the same letters. The galleries, *d d*, hold two large crucibles, *k k*, of cast iron; covered with clay luting in order that they may not adhere to the sulphide of antimony which flows into them. To each of these crucibles a set of wheels, running on parallel bars of iron, leading into the furnace, is affixed. In this way the crucibles are charged, placed in the furnace, and withdrawn with facility. Both galleries are covered with flat tiles of firebrick, *l l*, on which the conical fire-clay cylinders, *m m*, rest. The antimony ore for fusion is introduced into these fire-proof cylinders, and the product, purified by fusion, is received in the lined crucible beneath the cover, *l l*, through the connection, or small opening, *n*, at the base of the cylinders. *q q* is the arch of the furnace through which the cylinders ascend, by openings somewhat larger than their outer diameters: these orifices are closed at the top by clay covers, *r r*. The arch at the top, *s*, is constructed cylindrically, forming a double cross arch.

Fig. 8.



After the flame from the fire has played round the cylinders containing the mineral, it passes through three openings and flues into the chimney, *t*—Fig. 9. Two of these openings are indicated by the letters *u* and *v*, in this figure, which is a section of the elevation following the line, C D, of Fig. 7; these flues are provided with valves. *w* is a chimney for carrying off antimonious vapours which may be expelled from the cylinders while drawing out the slag and exhausted ore. Another chimney, *x*, begins above *y y*—see Fig. 8.—where the cylinders are charged; a partition, *z*, divides this chimney into two parts, so that the workman, while operating on one side, is not inconvenienced by the fumes of the other. *o o* are orifices several inches in height, by which the cylinders may be reached from the front and back part of the furnace by conical openings, such as *p*, in the walls; the openings, *o o*, are closed during the working with clay stoppers, *n n*, seen in the preceding figure, which are removed at the end of an operation. *a′* and *b′ b′* are iron and wooden beams and bands to brace the chimneys. *c′ c′* are conical arches on both sides of the furnace, closed with well-fitting plates, *d′ d′*; through these the interior of the furnace may be examined, and repairs made when required.

Fig. 9.



Each cylinder is charged with 490 lbs. of ore, previously warmed on the arch, the coarsest and richest of which is placed at the bottom of the cylinders. As soon as the melting of the ore is completed, the impurities remaining are withdrawn through the openings, *o o*, and a fresh charge is introduced. The trisulphide of antimony produced should have a bluish colour; if it possesses a reddish appearance, it is indicative that too great a heat has been applied. When the iron receivers are three-quarters full, they are drawn out and allowed to cool, and the lump of crude antimonious sulphide is removed. Fresh charges are put in every three hours, and about 1 cwt. of the trisulphide is obtained at each process, when the furnace is in good working order, On an average, the cylinders last about twenty days.

It is of extreme importance that the heat be not too great, since at a white heat antimony is completely volatilized. Nor should the ore be too finely pulverized, as in this case the melted sulphide cakes together with the gangue, and is very difficult to separate. The residues always contain from 10 to 12 per cent. of antimony, partly in the form of trisulphide, partly as trioxide.

The product of the preceding operations is next roasted in a reverberatory furnace to expel the whole of its sulphur, and to produce instead the trioxide of the metal; the latter is subsequently reduced by mixing it with about 20 per cent. of charcoal, saturated with a strong solution of carbonate of soda. According to Berthier, 70 per cent. of good metal may be procured from the ordinary product of fluxion of the crude sulphide, when mixed with 60 parts of iron scales from the hammer or rolling mill, 45 to 50 of carbonate of lime, and 10 to 12 of powdered charcoal, and the mixture heated to fusion in the ordinary way. Iron is often found in considerable quantities in the antimony procured from this mixture; and therefore, unless subsequently purified, it cannot, in many instances, be advantageously employed.

Uses in the Arts.—Antimony is chiefly used as an ingredient in making alloys, such as type-metal, Britannia-metal, plate-pewter, and various others. The typefounder uses two alloys of antimony, lead, and tin; the one composed of—

Tea lead, ...	75
Antimony, ..	20
Block tin, ..	5
	100

and the other

Tea lead, ...	70
Antimony, ...	25
Block tin, ...	5
	100

Stereotype metal is formed of

Tea lead, ...	112 lbs.
Antimony, ...	18 "
Block tin, ...	3 "
	133

The best Britannia-metal is composed of:—

Tin, ...	150 lbs.
Copper, ...	3 "
Antimony, ...	10 "

Common Britannia-metal:—

Tin, ...	140 lbs.
Copper, ...	3 "
Antimony, ...	9 "

Britannia-metal for Castings:—

Tin, ...	210 lbs.
Copper, ...	4 "
Antimony, ...	12 "

Britannia-metal for Lamps:—

Tin, ...	300 lbs.
Copper, ...	4 "
Antimony, ...	15 "

A higher proportion of antimony is sometimes used. Copper is added to give colour. The chief use of antimony in making stereotype plates is, that besides hardening them, it causes expansion of the alloy in cooling, and as the cooling next the type is latest, the expansion causes the metal to fill every interstice, and beautifully sharp casts result. Nearly every manufacturer uses different proportions of antimony according to the quality of his tin. The usual practice is to test a small quantity of the tin with antimony, before casting on the large scale.

Antimony imparts a peculiar beautiful red colour to copper, varying from rose red, with little antimony and much copper, to crimson and violet when the proportion of the metals more nearly approach equality. For this reason antimony is sometimes added to brass to improve its colour.

Antimony is used for the preparation of concave mirrors. It gives a finer texture to the mass, on which account it takes a better polish. It is also an ingredient of bell metal, being supposed to increase the clearness and strength of the sound, besides improving the colour of the bells.

From its easy fusibility, it is used to promote this quality in many alloys which are employed for artistic or manufacturing purposes.

Antimony forms compounds with oxygen, sulphur, and chlorine, in various proportions, as seen in the following table :—

Antimonious oxide (trioxide of antimony), ……	Sb_2O_3.
Antimonoso-antimonic oxide (tetroxide, or neutral oxide of antimony), ……	Sb_2O_4.
Antimonic oxide (pentoxide, or acid oxide of antimony), ……	Sb_2O_5.
Antimonious sulphide (trisulphide of antimony),..	Sb_2S_3.
Antimonic sulphide (pentasulphide of antimony),.	Sb_2S_5.
Antimonious chloride (trichloride of antimony),..	$SbCl_3$.
Antimonic chloride (pentachloride of antimony),..	$SbCl_5$.

It also unites with bromine, iodine, and selenium.

Of the preceding compounds, the trisulphide, trichloride, and trioxide, are the only important bodies in a manufacturing point of view. They are, with the exception of the trisulphide, which is employed to prepare the metal, almost exclusively used in medicine. These will now be described.

ANTIMONIOUS SULPHIDE.—*Trisulphide of antimony, anhydrous sulphantimonious acid, crude antimony, antimony ore, schwefelspiessglanz, anderthalb, schwefelspiessglanz rohes antimon*, German; *stibium sulphuratum nigrum, lupus metallorum*, Latin. Formula, Sb_2S_3.

Having already described the principal use of this antimonious compound, and the method followed for purifying it, nothing further remains but to add a few words on the manner in which the pure trisulphide is prepared for other purposes. The trisulphide hitherto described contains, even after careful fluxing, variable quantities of sulphides of iron, lead, copper, and arsenic; hence the black streak which the fused compound makes, instead of a blackish red, which is formed by the pure substance. Crystalline antimonious sulphide may be prepared by fusing 13 parts of pure antimony and 5 of flowers of sulphur (sublimed sulphur) in a crucible, the mixture being introduced by degrees and at short intervals. After the whole has been fused the crucible is taken from the fire and inverted, to allow the melted sulphide to flow out. The whole of the metal may not be decomposed in this operation, but those portions which have escaped the action of the sulphur are easily removed.

Pure trisulphide of antimony, as thus prepared by the first method, has a steel-grey colour, a shining metallic lustre, and a fibrous crystalline texture; it is brittle, and gives a reddish-black streak upon paper; it is very fusible, but less so than the metal, boils at a high temperature, and partially distils in a current of gas. When heated to whiteness in a crucible it loses from 10 to 12 per cent. of its weight of sulphur; heated in the air, it suffers decomposition, giving off its sulphur in the form of sulphurous acid, and absorbing oxygen, which converts it into trioxide of antimony.

A current of hydrogen gas passed over it at a low red heat completely desulphurizes it, leaving pure metal. This property of hydrogen, that of depriving it of sulphur at a red heat, is possessed also by many metals, of which the most energetic is iron. When exposed with charcoal to a white heat, the antimonious sulphide is likewise reduced to metal.

When the trisulphide and trioxide of antimony are fused together, combination takes place, without either of the compounds suffering decomposition.

Its chief use, besides its employment as a source for the production of the metal, is for pharmaceutical preparations, chiefly tartar emetic.

Amorphous Trisulphide of Antimony; Kermes Mineral; brown-red sulphide of antimony; Pulvis carthusianum, sulphur stibiatum rubrum. — Amorphous trisulphide of antimony may be made by various processes. For instance, by conducting a stream of sulphuretted hydrogen gas through a concentrated solution of tartar emetic (potassio-antimonious tartrate, $K(SbO)C_4H_4O_6$), an orange-red precipitate is formed, which is the sulphide in question. When *kermes mineral* is digested for some time with tartaric acid, any antimonious oxide or potassium salt which may be present is removed, and pure trisulphide of antimony remains. Again, by digesting a mixture of 1 part of crude trisulphide of antimony with 1 part of pearl ash (crude potassium carbonate), 1½ of slaked lime, and 15 of water, in a closed vessel for two hours. During the digestion a soluble double sulphide of antimony and alkali is formed; and on adding sulphuric acid to decompose the alkaline sulphide, sulphide of antimony falls down; this precipitate is then filtered, washed with sulphide of hydrogen water, and dried.

FUCHS obtained pure amorphous sulphide by fusing the grey trisulphide of antimony, and then cooling the vessel suddenly by immersion in cold water. LIEBIG, as will be seen in a subsequent process, converts the crystalline native sulphide into the amorphous variety by dissolving it in an alkaline ley, and precipitating by an acid; or by igniting 1 part of antimony with 2 parts of a mixture of 1 part nitre with 2 parts cream of tartar, extracting with water, filtering whilst hot, and precipitating the filtrate with an alkaline carbonate.

Mineral kermes varies in composition according to the process by which it is prepared. Some of the processes yield pure trisulphide of antimony, others a compound of antimonious sulphide and oxide, whilst that investigated by BERZELIUS and ROSE contained sulphantimonite of potassium or sodium.

The oldest method of making mineral kermes is that described by LA LIGÉRIE. The grey sulphide is powdered finely, and then boiled with an alkaline carbonate, and the filtered solution allowed to cool. This method is still given in the Prussian Pharmacopœia. The crystalline grey sulphide is much less soluble in alkaline carbonates than the amorphous precipitated red sulphide. LIEBIG therefore recommends that 1 part of finely powdered grey sulphide should be boiled with 1 part of potassium hydrate and 30 parts of water for one hour, and the filtrate precipitated while still hot by dilute sulphuric acid. The mixed salts are then divided into 3 parts, each of which is washed with water, in a separate vessel, by subsidence and decantation, and afterwards collected on a filter. The precipitate collected on the first filter is then thrown by successive portions into a boiling solution of 1 part of dry carbonate of soda in 34 parts of water, and the whole boiled for an hour; and the solution (which need not be filtered, since nothing remains undissolved) is then slowly cooled to separate the kermes. The solution poured off from the precipitated kermes is again raised to the boiling point, and the contents of the second filter added to it, and similarly with the third filter. The kermes deposited after the second boiling has usually the finest colour. The kermes thus prepared amounts, after washing with cold water and drying, to half the quantity of the grey sulphide used. It must be regarded (according to Liebig) as $Sb_2O_3 2Sb_2S_3$ (a composition similar to that of red antimony ore), but it still contains from 1 to 1·5 per cent of soda.

Kermes free from antimonious oxide may be prepared by the following process:—A mixture of 1 part of finely powdered trisulphide of antimony, with 1 part of potassium carbonate, 1·5 part of potassium hydrate, and 15 parts of water, is digested in a close vessel for two hours; the filtrate diluted with a large quantity of water, and precipitated by sulphuric acid; the precipitate boiled with dilute sulphuric acid (to decompose the potassium sulphide present), and the residue washed with water.

H. Rose gives two processes. The first is to heat slowly 1 part of pure potassium or sodium carbonate and 2¾ parts of grey sulphide of antimony, in a covered crucible, till the mass fuses. This mixture is boiled with water, and filtered; the compound sulphide dissolves out and, on exposing the solution to the air, precipitates as the liquid cools. After the first filtration the residue is heated with water, or with the clear mother liquid left after a previous deposit of kermes; a further quantity of the compound is dissolved out, which separates from the liquid, as before, as soon as the whole becomes cold. When the boiling solution has been thus treated three or four times in succession, there remains an insoluble substance, consisting mainly of a trisulphide and trioxide of antimony, Sb_2S_3,Sb_2O_3.

The quantity of kermes thus obtained is much greater than that produced by boiling grey sulphide of antimony with carbonate of soda, because more sulphide of antimony is decomposed by fusion; but the kermes has more of a yellowish brown colour, and contains a larger amount of antimonious oxide (Sb_2O_3), though in variable proportions. The more rapidly the kermes is collected in the filter after cooling, the less is the quantity of oxide mixed with it. Besides antimonious oxide, the kermes also contains a compound of potassium or sodium sulphide, with pentasulphide of antimony (K_2S,Sb_2S_5), potassium sulphantimonate. Hence such a kermes, if prepared with sodium carbonate, yields sodium chloride (about 8 per cent.) when decomposed by chlorine gas. And the liquid from which the kermes has been deposited contains carbonate of soda, sodium sulphantimonate (Na_2S,Sb_2S_5), and sulphantimonite (Na_2S,Sb_2S_3). On evaporation it yields—first, crystals of sodium antimonate, and then of carbonate of soda. Addition of sodium bicarbonate decomposes the sulphantimonite remaining in solution, and brownish red kermes are deposited. Duflos remarks that the larger the proportion of alkaline carbonate, and the longer the fusion is continued, the smaller the quantity of antimonious oxide mixed with the kermes; but if too much alkaline carbonate is used no kermes is deposited. But if carbonic acid gas be passed through the solution, a brilliant kermes is precipitated in large quantity; and when the carbonic acid ceases to cause any further precipitation, the stronger acids, after expelling the carbonic acid, throw down pentasulphide of antimony (Sb_2S_5) from the liquid filtered from the kermes. Moreover, if the liquid precipitated by carbonic acid and filtered from the kermes be boiled with a fresh quantity of trisulphide of antimony, it deposits kermes on cooling, because the solution then contains carbonate of potash.

Another method is to fuse 2 parts of metallic antimony, 1 of sulphur, and 3 of carbonate of soda; or 16 parts of antimony, 3 of sulphur, and 32 parts of potassium carbonate; or to heat a mixture of from 3 to 4 parts of cream of tartar (potassium acid tartrate) ($KC_4H_5O_6$), and 1 part of grey sulphide of antimony, in a crucible, till the vapour of decomposed tartaric acid ceases to be given off.

The kermes which separates after boiling the crude antimony with alkaline carbonate in excess, and cooling the liquid, contains trioxide of antimony. After drying the precipitate the antimonious oxide becomes very apparent, especially if examined with the microscope, which shows its definite crystalline structure, interspersed with the amorphous flocculi of the kermes.

Kermes mineral was at one time in high repute on the Continent as a medicine, and indeed is still occasionally used in considerable quantities; in England it is largely used as a veterinary medicine. Pereira states that, when administered in small doses, it acts as a sudorific and liquefacient; in large doses, as an emetic and purgative. The uncertainty of its operation is, however, a great drawback; even when moderate action follows its use, it is doubtful whether it has any advantage over tartar emetic.

Hydrochloric acid decomposes kermes mineral with the formation of the trichloride, sulphuretted hydrogen being at the same time evolved and sulphur precipitated.

Pentasulphide of Antimony.—*Antimonic sulphide, anhydrous sulphantimonic acid, golden sulphide of antimony, sulphur antimonii auratum* (Sb_2S_5).—This body is an amorphous yellowish-red powder, having a sweetish sulphurous taste and odour. It burns with a pale blue flame when heated in the air; heated in a closed vessel it is decomposed into trisulphide and free sulphur. When exposed to moist air it is wholly decomposed, sulphuretted hydrogen is evolved and trioxide of antimony formed. It is completely soluble in hydrochloric acid with the evolution of sulphide of hydrogen, a solution of trichloride of antimony being formed. When boiled with a solution of cream of tartar, tartar emetic is produced from the solution of the oxide of antimony contained in the precipitate. Its colour distinguishes it from the sulphur compounds already mentioned.

The commercial pentasulphide is often of a very bright colour; it is prepared by boiling the amorphous trisulphide with potassium hydrate and ground sulphur, and after filtering off the liquor, precipitating by means of an acid in the usual way Or, 4 parts of the trisulphide of antimony are boiled with 8 of lime and 80 parts of water, the liquor strained and precipitated by hydrochloric acid in the usual way. Or, 2 parts of the trisulphide may be fused in a crucible with 4 parts of carbonate of potassium and 1 of sulphur; the residue, when cold, boiled with 20 parts of hot water; the solution filtered and decomposed by a large excess of sulphuric acid.

The medicinal effects of this compound are more active than those of the kermes, on account of the larger proportion of teroxide of antimony which it contains. Its uses are as an alterative in chronic diseases, particularly cutaneous affections, glandular enlargements, rheumatism, and liver complaints.

Sulphantimonites.—Trisulphide of antimony combines readily with basic metallic sulphides. Many of these bodies are found as minerals, such as—

Zinkenite, ...	Pb_2S,Sb_2S_3
Myargyrite, ...	Ag_2S,Sb_2S_3
Antimonial copper glance, ...	Cu_4S,Sb_2S_3
Jamesonite, ...	$(Pb_2S)_3Sb_2S_3$
Feather ore, ...	$(Pb_2S)_2Sb_2S_3$
Stephanite, ...	$(Ag_2S)_6Sb_2S_3$, &c.

Livers of Antimony.—These are the artificial sulphantimonites of the alkali metals. They result from the fusion of trisulphide of antimony with alkaline carbonates.

Pentasulphide of antimony is a strong sulphur acid. It forms salts with most of the basic metallic sulphides, which have the composition $(M_2S)_3Sb_2S_5$ or M_3SbS_4.

ANTIMONIOUS OXIDE. *Trioxide of antimony* (Sb_2O_3), *valentinite, white antimony, antimony bloom.*—This substance is found as a mineral. It occurs in veins of the primary rocks in Bohemia, Saxony, and Hungary, in shining white crystals of the trimetric system, and also in Algeria in regular octahedra. The artificial crystalline trioxide is likewise found to be dimorphous. This compound was known to BASIL VALENTINE, who called it *flores antimonii.* In commerce it is termed indifferently *protoxide*, *sesquioxide*, and *flowers of antimony.*

It may be prepared in several ways. 1. On heating metallic antimony to the point of its combustion it burns with a bluish light, and if the crucible be reclined to one side to admit a current of air, small acicular crystals of trioxide will be observed on the upper part. 2. By adding metallic antimony in powder to sulphuric acid, evaporating the liquid to dryness, and then washing the dry mass, first with water and next with a weak solution of carbonate of soda, for the purpose of removing the last portions of sulphuric acid; after completely washing the residue a white powder remains, which is trioxide of antimony. 3. By digesting finely-ground grey sulphide of antimony in about four times its weight of hydrochloric acid, trichloride of antimony is formed and sulphide of hydrogen escapes with effervescence, the former of which is decomposed by carbonate of soda into trioxide of antimony, which precipitates, while carbonic acid is liberated, and the soda is converted into chloride of sodium by the chlorine of the antimonial compound.

The precipitated trioxide is collected upon a filter, washed well with water, and dried. For pharmaceutical purposes the Dublin college recommends the treatment of 20 parts of the powdered grey sulphide of antimony with 100 parts of hydrochloric acid and 1 of nitric acid; the acid and powder must be slowly brought together, and a gentle heat applied for some time, which may be afterwards increased, and the solution left to digest as long as any effervescence or escape of sulphuretted hydrogen is observed; and then the whole is to be boiled for an hour, the liquor filtered when cold, and the filtrate received in a vessel containing 1 gallon of cold water, which precipitates the basic trichloride of antimony. The oxychloride is collected, and washed with water till the washings cease to evince an acid reaction with litmus paper. During the washing of the precipitate the water is decomposed, its oxygen being retained by the antimony and antimonious oxide (Sb_2O_3) formed, hydrochloric acid being given off.

DUMAS directs the preparation of trioxide of antimony to be conducted as follows:—Heat pulverized antimony in a shallow vessel exposed to the air; when the oxidation has advanced, the metal takes fire and becomes incandescent, antimonic oxide (Sb_2O_5), mixed with some of the metal, being produced; the whole of the compound is now transferred to a crucible, in which it is heated to fusion, when the antimonic oxide is reduced to the state of trioxide by the excess of metal, which is found, with the fused mass, in a button at the bottom.

Trioxide of antimony is a white powder, fusible, and volatile at a red heat; when heated in close vessels it suffers no change, but is partly sublimed, and condenses in acicular or octahedral crystals on the cover, or upper part of the pot.

If the trioxide be kept in fusion, with contact of air, it is converted into pentoxide (antimonic oxide, Sb_2O_5); but if the air be excluded, it concretes into a silky crystalline mass, presenting a greyish appearance.

Water dissolves oxide of antimony in small quantity. Nitric acid and chlorate of potassium rapidly convert the trioxide into pentoxide; sulphur decomposes it in the heat into trisulphide, with the formation of water; and when heated to redness with charcoal, metallic antimony is obtained. Tartaric acid readily dissolves the antimonious oxide; acid tartrate of potassium acts similarly. In each case tartar emetic is produced.

TRICHLORIDE OF ANTIMONY.—*Butter of Antimony* ($SbCl_3$) is used to some extent in medicine, chiefly as a caustic.

On dissolving sulphide of antimony in hydrochloric acid, as before explained, trichloride of antimony is formed, with the evolution of sulphuretted hydrogen.

The action of the hydrochloric acid is greatly accelerated by mixing with it a small proportion of nitric acid. When the solution thus obtained is evaporated, the trichloride of antimony remains in the solid state. To prepare the pure salt, the hydrochloric acid solution of the trichloride should be evaporated in a porcelain dish, till it begins to crystallize on the dish being placed in a cold situation; the concentrated liquid is then transferred to a retort, and distilled until a drop of the liquid solidifies upon a cold surface; at this point the receiver is changed, and the remaining portion of the charge collected as pure.

It is white, semitransparent, crystalline, of a buttery consistence, and deliquescent in the air.

When added to water it gives a bulky white precipitate of powder of algaroth, which consists of trioxide, united with a portion of the trichloride, while hydrochloric acid remains in the solution.

Trichloride of antimony affords, with all the other reagents, similar reactions to those which distinguish the other salts of this metal.

Besides being used as a caustic, it is employed in the arts, especially by gunmakers, for the purpose of giving a yellow colour to gun barrels, and is called *bronzing salt*. It is used for this purpose in the state of solution, mixed with olive oil, and rubbed over the iron slightly heated, which is then exposed to the air till the desired colour is obtained. Sometimes, to quicken the effect, nitric acid is added. When bronzed, the surface of the iron is polished by a burnisher, or by wax, or by varnish composed of 2 ounces of shell-lac and 3 drachms of dragon's-blood dissolved in 2 quarts of spirit of wine.

Quantitative Estimation of Antimony by Analysis.—The percentage of antimony in a mineral may be estimated from the amount of the trisulphide of antimony, the tetroxide or antimonate of antimony (Sb_2O_4 or SbO_2), or of the metal which it produces when operated upon with suitable reagents.

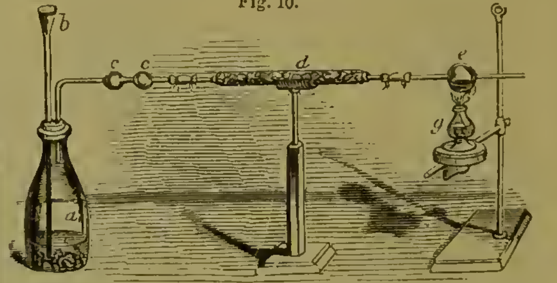
As Trisulphide.—A solution of the metal is obtained by treating the ore with strong hydrochloric acid, containing some nitric acid (if silver be absent). This solution is diluted with water, and should any turbidness ensue, tartaric acid must be added to dissolve it; or if the addition of this acid would interfere with the estimation of any other metal, hydrochloric acid is employed to effect the same purpose. Sulphuretted hydrogen is then transmitted through the liquid, until the solution acquires a strong odour of the gas, when the whole of the antimony will be converted into the red trisulphide. If antimony is the only metal present, the vessel holding the precipitate may be placed on a hot sand-bath till the excess of sulphuretted hydrogen is expelled. By this means the small quantity of trisulphide retained in solution by the excess of the gas is set at liberty. The solution is then filtered through a dried and tared filter, washed with water, dried in a water bath till the weight remains constant, and weighed.

As Tetroxide.—As the antimony in solution is often in a higher state of oxidation than the teroxide, especially if nitric acid has been employed, and as more or less of the sulphide of hydrogen is decomposed by the free acid of the solution before precipitation, the sulphide of antimony produced contains an excess of sulphur. In this case the excess of sulphur is to be extracted with carbon disulphide, and the antimony trisulphide introduced into a porcelain crucible, and then add—first, a few drops of strong nitric acid, and then fuming nitric acid in quantity about ten times the amount of the precipitate, and allow the acid to evaporate gradually in the water bath. The sulphur separates at first as a white powder, but is completely oxidized during the evaporation. The white residue is antimonic oxide with sulphuric acid, and may be converted into the tetroxide (Sb_2O_4) by ignition.

As the tetroxide is neither volatile nor decomposable by a red heat, it affords a very accurate means of determining antimony quantitatively. The Sb_2O_4 contains 79·22 per cent. of the metal.

As Metal.—Having ascertained the weight of the dry trisulphide produced by treatment with acids, and precipitating by sulphuretted hydrogen in the way already pointed out, a known quantity is to be taken and decomposed by heating in an atmosphere of hydrogen; sulphuretted hydrogen is evolved, and metallic antimony is left. A convenient apparatus for this purpose is seen in Fig. 10. The flask, *a*, contains water and a few bars of zinc; sulphuric acid is poured in through the funnel, *b*, in order to generate the gas, which escapes by a tube opening just below the cork of the flask, and which is provided with two bulbs, *c c*, where the greater part of the moisture carried away by the gas is condensed. To dry the hydrogen completely, it is conducted through another large tube, *d*, filled with fragments of chloride of calcium, and thence into the bulb, *e*, holding the substance to be reduced.

Fig. 10.



Before commencing the operation, the bulb, *e*, with its glass tubes attached, should be thoroughly cleaned, dried, and weighed, and the trisulphide of antimony then introduced. The bulbed tube and its contents are now reweighed, and the increase of weight is the quantity of the compound submitted to experiment. The various parts of the apparatus are next well connected by means of caoutchouc tubes, as seen in the figure, and the hydrogen generated; as soon as the apparatus is filled with the gas, a gentle heat is applied by means of a spirit-lamp, *g*, to the bulb containing the substance, till the reduction is completed. If the compound consists of trisulphide of antimony only, scarcely any sulphur is sublimed during the reduction, as it passes off in the form of sulphuretted hydrogen; but should the pentasulphide be present, sulphur will be sublimed at the same time that the remaining part unites with the hydrogen and escapes. The sublimed sulphur is expelled by bringing the heat of the lamp gradually towards that part of the tube where it is deposited. When it is observed that no more sulphur condenses in the tube, and that sulphuretted hydrogen ceases to be evolved from the extremity, the apparatus is allowed to cool, but the current of hydrogen is still maintained for some time. As soon as the apparatus is perfectly cold, it is taken to pieces, the bulb-tube, *c*, wiped clean and weighed; the difference between the weight of the bulb-tube and substance before the experiment, and that which is now obtained, is the amount of sulphur in the antimony sulphide reduced, and the difference between the weight of the empty tube and its weight after the reduction, is the weight of the metal yielded by the sample operated upon.

Considerable care must be exercised in this reduction to arrive at accurate results: since it is almost impossible to prevent the sublimation of some of the antimony, if the heat applied be at all high; and on the other hand, if the temperature be too low, if it be too feebly applied, part of the sulphur remains without being expelled.

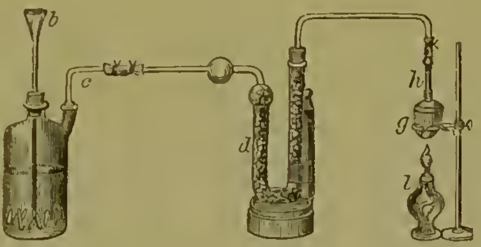
When antimony is alloyed with other metals, or associated with them in mineral substances, its amount may be determined, except when united to tin, by dissolving the substance in nitric acid, or in aqua regia containing an excess of nitric acid. The antimony is converted into trioxide of antimony, which precipitates, while the other metallic oxides are dissolved. Before the whole of the antimony is thrown down, however, it is necessary to decompose any trichloride that may be formed (when aqua regia has been used as the solvent), by evaporating the liquid with an excess of nitric acid. The residue is then thrown upon a filter and washed with water, dried, and its weight determined, after which a portion is taken and converted into tetroxide, or metal, as already described.

This method does not yield strictly accurate results, on account of the precipitated trioxide of antimony not being perfectly insoluble in nitric acid, and, therefore, the filtrate and washings always contain some traces of antimony; yet, for technological purposes, the results obtained are sufficiently correct.

H. ROSE reduces the tri- and penta- sulphides of antimony in the apparatus given below, fig. 11. The hydrogen generator, *a*, contains zinc and water; *b* is a funnel-tube for adding sulphuric acid; *c*, a tube conveying the gas to the chloride of calcium tube and bulb, *d*, where it is divested of moisture. The gas, after leaving the chloride of calcium tube, passes to the crucible, *g*, containing the body to be reduced, through a small porcelain tube, *h*. Before the reduction commences, the crucible with the lid and its tube is balanced, and then the substance introduced, and the whole reweighed; the difference in the two weighings is that of the body taken. All the connections of the apparatus are now made air-tight, and the crucible attached; hydrogen is generated in the bottle, *a*, by pouring sulphuric acid down the funnel tube, *b*, and when the whole apparatus becomes filled with the gas, heat is applied by means of a spirit-lamp, *l*, to the crucible, taking all the precautions previously mentioned. When it is observed that no more sulphuretted hydrogen is evolved from the end of the tube, the heat is removed, but the current of hydrogen is maintained during the cooling of the apparatus, after which it is taken asunder, and the crucible with its contents weighed.

Arsenic being frequently associated with antimony in its ores and alloys, NOAD suggests the following method for their separation:—Having dissolved the ore or alloy in aqua regia, sulphides of antimony and arsenic are obtained by saturating the solution with ammonia, and then adding ammonium sulphide containing an excess of sulphur; the whole is digested for some time, then filtered, and hydrochloric acid added to the filtrate in slight excess, to precipitate the dissolved sulphides. The precipitate is collected upon a dry tared filter, washed with water impregnated with a little sulphuretted hydrogen, and dried in the water-bath till all moisture is expelled, after which it is weighed.

Fig. 11



A portion of the mixed sulphides is then introduced into the bulb tube, Fig. 10, and a stream of hydrogen gas passed over it, while a proper heat is applied by the lamp; all the trisulphide of arsenic (As_2S_3), with the excess of sulphur, is expelled, leaving trisulphide of antimony, which is to be weighed.

DRY ASSAY OF ANTIMONIAL ORES.—All the ores of antimony, for the purpose of assay, may be divided into two classes: those in which the metal is combined with oxygen or chlorine, and in which little or no sulphur is present; and compounds of sulphur and the metal.

All the ores belonging to the first class are very easily reduced (provided no earthy or silicious matters are present), by simply heating them to redness with finely-divided charcoal. The assay may be conducted in an earthen crucible; but as antimony is readily volatilized, care must be taken that the heat is not too high during the reduction. If the ore contain calcareous or silicious impurities, it is necessary to mix it either with 3 parts of black flux, or 1 of carbonate of soda, and $\frac{1}{4}$ part of charcoal finely ground. When the mixture is in a tranquil state of fusion, the crucible is to be withdrawn and gently tapped, to cause the small globules of antimony to unite, and form one button at the bottom.

The crucible is broken as soon as it gets cold, and the metallic button at the bottom carefully freed from adhering matter, by gently tapping it with the hammer, brushing off dust or other adhering matter, and weighed. Unless great delicacy of manipulation be observed when detaching the slag, portions of the metal will be removed on account of the brittleness of the antimony rendering it liable to the detachment of small particles, which, of course, would be so much loss.

Those ores which consist principally of trioxide of antimony, but which contain small quantities of sulphur, may be analyzed by the foregoing method; for as the sulphide yields to the black flux just half its combined antimony, a very small portion only can be retained in the slag.

Ores of the second class offer more difficulties than those just described. Their assay may be performed either by first roasting the sulphide, and subsequently fusing the oxide produced with black flux, or by directly treating the mineral, reduced to an impalpable powder, with finely-divided metallic iron or iron scales, and a little black flux, at an elevated temperature.

As the grey sulphide of antimony is very fusible and volatile, it requires much care and attention to roast it thoroughly, in order to expel the sulphur, and form the trioxide of the metal; to be successful, the temperature should be moderate, and the mass kept stirred with an iron rod or wire as long as any sulphurous acid is given off. It is then fused with 3 parts of black flux in the usual way, and a button of antimony is obtained.

The results of the dry assay of antimony ores are so very fallacious, that the process is now but seldom used.

ARSENIC.—*Arsenic*, French; *arsenik*, *arsen*, *scherbenkobalt*, *fliegengift*, *näpchenkobalt*, German; *arsenicum*, *regulus arsenici*, Latin; ἀρσενικὸν, Greek. Symbol, As; atomic weight, 75.

Arsenic is mentioned by DIOSCORIDES and other authors who lived about the commencement of the Christian era. In their works the word was used to signify a reddish mineral composed of arsenic and sulphur, which was used as a medicine and in painting.

Arsenic is found native, but more often in combination with other metals and sulphur.

The distinct metallic characters of arsenic were discovered by BRANDT in 1773, who gives the first accurate process for procuring it. He mixed white oxide (arsenious oxide or arsenious acid (As_2O_3) with potassium hydrate and ammonium chloride, and fused the mixture in a well-luted crucible. Its properties were further investigated by MACQUER in 1746, by MUNNET in 1773, and by BERGMANN in 1777; but the latter took many of his facts from BRANDT's paper.

Arsenic forms two native compounds with oxygen, namely, arsenious and arsenic acid. Orpiment and realgar, two of its sulphides, are also met with in the mineral kingdom. It has been discovered in minute quantity in a large number of mineral waters. Small quantities of arsenic are always found with iron ores, and frequently influence their quality. It is often found in combination with metallic sulphides, especially with the sulphide of iron, constituting arsenical pyrites ($FeAs + FeS_2$); and two kinds of arsenical iron are found, having the compositions FeAs and Fe_4As_3 respectively.

Arsenic is obtained as a bye-product from arsenical cobalt, cobalt glance, bismuth cobalt ore, nickel ochre, polybasite, red silver, arseniate of copper (of which several varieties are known), from euchroite, kupferschaum, erinite, scorodite, smaltine, cloanthite, nickel glance, copper nickel, arsenical nickel, arsenical fahl ores, &c.

The principal mines are those of Freiberg, Annaberg, Marienberg, and Schneeberg; Joachimsthal in Bohemia, Andreasberg in the Harz, Oravicza in Hungary, Kongsberg in Norway, Zmeoff in Siberia, where native arsenic is found in large masses, and at St. Marie-aux-Mines in Alsace.

The metal is readily prepared in the laboratory by mixing 1 part of pure arsenious acid with 3 of charcoal, or a mixture of charcoal and sodium carbonate in a crucible, on which another of a similar shape is inverted as a head, and applying a moderate heat, either in a furnace or over a lamp. By keeping the cover as cool as possible the metal will be found in the course of a short time to have sublimed, and to be adhering to the interior of the crucible in the shape of a brilliant metallic coating.

Arsenic is of a bluish-white colour, approaching steel grey, and has a crystalline structure. It is the most volatile of all metallic bodies, being completely vaporized without fusion at 365° Fahr. (185° C.). It is so brittle that it may be easily reduced to a fine powder by trituration in a mortar. Kept in water it suffers no alteration; but when exposed to the air it soon loses its lustre, becomes black, and falls to powder. When exposed to a moderate heat in free contact with air it sublimes in the form of a white powder, and at the same time emits its characteristic alliaceous odour, and is wholly converted into arsenious acid (As_2O_3). With limited access of air metallic arsenic is deposited in a compact brown mass, having a strong metallic lustre. If air be excluded and the temperature be high, the arsenic is deposited as a nearly white crystalline mass, which does not oxidize in the air even when heated to 80° C. The other forms readily pass into arsenious oxide by exposure to air.

Arsenic burns in air or oxygen with a pale blue flame into arsenious oxide (As_2O_3).

ORFILA states that metallic arsenic, when swallowed, acts as a powerful poison, probably by being oxidized into arsenious acid.

The specific gravity of arsenic is between 5·62 and 5·96, its vapour density is 10·3995, air being unity: or 150, hydrogen being 1.

Heated with chlorine, arsenic ignites spontaneously, producing a brilliant white flame and forming arsenious chloride ($AsCl_3$). It is soluble in nitric acid and in aqua regia, and is converted by this treatment into arsenious or arsenic acid, according to the more or less prolonged action of the solvents. It enters into fusion with most of the metals, and by this union renders those which were malleable more brittle, and those before difficultly fusible more easily melted; it also confers a brightness upon the alloy, which often renders its presence in small quantities desirable.

The metal is prepared on the large scale by subjecting arsenical pyrites (Fe_2AsS_2) or arsenical iron ($FeAs + Fe_4As_3$) to sublimation in earthenware retorts, to which are attached receivers, wherein a piece of rolled sheet iron is generally placed, which collects the whole or greater part of the sublimated metal.

Arsenic forms two oxides—trioxide, anhydrous arsenious acid or arsenious oxide, As_2O_3, and pentoxide, anhydrous arsenic acid or arsenic oxide, As_2O_5.

Arsenious Acid.—*Arsenious anhydride*, *Arsenious oxide*, *Anhydrous arsenious acid. Acide arsenieux*, French; *arseniksäure*, German; *acidum arseniosum*, Latin. *White arsenic*, *white oxide of arsenic*. Formula. As_2O_3.

This compound is formed, as previously stated, when arsenic is heated in the open air; it sublimes in white fumes, and when collected constitutes arsenious acid. It is usually in the form of a compact white cake; but it may also be obtained crystallized in octahedra by cooling the vapour so quickly that it solidifies at once without becoming semiliquid. It has an acrid taste, which leaves an impression of sweetness; but it is one of the most virulent poisons known. Arsenious acid occurs native as Arsenite or Arsenolite.

Fig. 1.

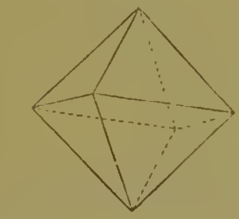
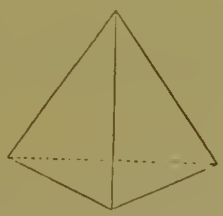


Fig. 2.



Arsenious acid is soluble in boiling water to a moderate extent, but considerably less so in cold; it is also soluble in alcohol and oils. Regarding the solubility of arsenious acid, Guibourt found that the crystallized acid required 104·17 parts of water at 60° Fahr. (15°·5 C.), and the amorphous 80 parts. Boiling water dissolved 9·68 of the former, and 11·47 parts of the latter; the solutions, upon being cooled at 60°, retained 1·78 of the crystalline, and 2·9 of the amorphous. Both solutions communicate a feeble acid reaction to blue litmus paper.

Arsenious acid is found in two distinct conditions—crystallized and amorphous. The crystallized acid often assumes two forms; the octahedron and tetrahedron.—Figs. 1 and 2. Wöhler has observed that the acid assumes the form of hexahedral plates, derived from the right rhombic prism; hence it appears to be dimorphous, which seems connected with the peculiarities of the opaque and vitreous state of the acid.

Vitreous arsenious acid, when recently prepared, is in the form of large, glassy, colourless, transparent cakes; it is sometimes of a yellowish colour, and in concentric laminæ formed by successive sublimations. By exposing these transparent cakes to the air, they are readily covered with a white coating, and lose their former transparency. This change is gradually extended to the centre, so that the fracture has an enamel-like appearance; occasionally the cakes crumble down into a friable mass. The reason of this change has been ascribed by Kruger to the absorption of water, as, according to that chemist, no change takes place in perfectly dry air; he ascertained that the acid increased in weight, though not more than $\frac{1}{100}$th of the whole mass.

Pereira kept arsenious acid in a sealed tube for two years without any change being noticed, but on cracking the tube the transparency of the inclosed acid was in a very short time lost.

A singular property of arsenious acid observed by Rose is, that when the vitreous acid is dissolved in hydrochloric acid, and allowed to cool slowly, vivid flashes of light are emitted from the crystals as they form. The phenomenon does not attend the crystallization if the opaque acid is dissolved in the acid liquor, neither will it succeed if the crystals deposited from the vitreous substance be redissolved in the acid, and suffered to cool as they acquire the property of the opaque acid; the production of the light seems to be connected with the transition from one modification to the other.

Heated on charcoal before the blowpipe, arsenious oxide emits the peculiar characteristic arsenical odour; when mixed with carbonate of soda and charcoal, and the compound subjected to heat in a glass tube, it yields a ring of metallic arsenic which condenses on the cold part of the tube. Arsenious acid is decomposed at an incipient red heat by hydrogen, carbon, and many of the metals.

Sulphuretted hydrogen produces, in solutions of arsenious acid, a lemon-yellow precipitate of trisulphide of arsenic (As_2S_3), which is very characteristic of this body, the yellow tint being observed when $\frac{1}{10000}$th of the acid is present, and a precipitate becoming visible in an acidulated solution of 1 part of arsenious acid in 80,000 parts of water. Excess of lime-water occasions a white precipitate in a liquid containing about $\frac{1}{500}$th of arsenious acid. Ammonio-sulphate of copper gives an apple-green precipitate in a solution of arsenious acid, thus indicating about a $\frac{12}{1000}$th part of the acid; and according to Reinsch, when a slip of bright copper leaf is boiled in an aqueous solution, acidulated by hydrochloric acid, a grey film of arsenic is deposited upon the copper, showing the presence of less than $\frac{1}{100,000}$th part of the acid. Nitrate of silver gives with it a yellow precipitate.

Arsenious acid unites with most metals, with which it forms salts, called *arsenites*. With oxide of copper it gives compounds much used as pigments, all which will be hereafter described.

Manufacture of White Arsenic, or Flour of Arsenic.—This is the arsenious acid in the form of a powder, white and fine as flour. The modes of its preparation in England and on the Continent somewhat differ. In England its manufacture is carried out in Cornwall and Devon, and it is also produced at Swansea. From the Mineral Statistics of the United Kingdom for 1873, we learn that in that year the quantity made was 5448 tons, of a value of £22,854, the greater part of what was thus returned being crude arsenious acid. Since then the manufacture has been stimulated by a larger demand and increased price; the refined product which in 1873 was sold at £6 8*s.* 9*d.* per ton, has since been sold at £15 per ton, and even quoted at £18 to £20 per ton.

The only mineral from which in this country arsenious acid is in any quantity prepared, is arsenical pyrites or mispickel, the formula of which is $FeAs + FeS_2$; this gives 46·58 per cent. of arsenic, while by analysis about 43 per cent. is obtained. This arsenical pyrites is associated with various other metallic minerals, the presence of which requires special attention. Thus, ores are raised which may contain any or all of the following:—Arsenical pyrites, iron pyrites, copper pyrites, tinstone, wolfram, blende, and galena, besides quartz and other non-metallic minerals. It will depend on its associates whether the arsenic will be the main end of the processes, or whether it shall be counted but a bye-product. The same circumstances will determine what shall be the preliminary mechanical preparation of the ore. Thus, where tinstone is present in any quantity that will repay its separation by the mechanical means always employed for that purpose—in a proportion, it may be, of less than 1 per cent. even—the ore is stamped to a fine state of division, and undergoes certain of the washing processes peculiar to the preparation of tin ore before it is considered fit to be treated for arsenic, and afterwards the residue is again mechanically treated for tin. Where there is a mixture of copper ore and arsenical pyrites, the material is generally separated by eye and hand into two classes—one from which copper will afterwards be extracted, and one which contains so little copper that its extraction would not be profitable; the only difference in their treatment is in the destination of the residue, that of the latter class being considered as refuse. These copper and arsenic ores are crushed between rolls, and may be sent through a sieve with apertures of 1¾ inch or less.

The percentage of arsenic in the ores treated varies much. At one extreme are those tin ores for which calcining is necessary for the sake of the tin, and of which the arsenic is but a bye-product; at the other is ore which contains little but quartz and arsenical pyrites, the latter in quantity that will well repay extraction. In this class, and in ore which contains both arsenic and copper, the arsenical pyrites may vary from 12 to 30 per cent. It is useful to be able to determine the percentage of arsenic present in the ore, readily if roughly. At many mines a very rough and ready plan is followed:—100 or 200 grains of the ore is heated over the fire in an iron ladle till all the arsenic and sulphur are burnt away; the weight of the residue is then compared with the original weight, and from the difference the richness of the ore is judged. The proportion of pyrites other than arsenical in the ore having previously been estimated by eye, an allowance for this is made; but, as usually done, this allowance and the other calculations are incorrect. It may therefore be useful here to put down, for the advantage of those who will adhere to this system rather than attempt a complete chemical determination of the arsenic, the true percentages of loss on roasting the three kinds of pyrites—copper, iron, and arsenical—or as they are termed in Cornwall, yellow ore, sulphur mundic, and white mundic.

Copper pyrites loses on roasting, ...	14 per cent.*
Iron pyrites loses on roasting, ...	33 "
Arsenical pyrites loses on roasting, ..	51 "

Also, by theory, a loss of 100 in the weight of arsenical pyrites implies the formation of 119 of arsenious acid or white arsenic.

If, therefore, for an example, an inspection shows the ore to contain, besides non-metallic substances, the three minerals about in the proportions 1, 2, and 4; then 14 × 1 (= 14), 33 × 2 (= 66) and 51 × 4 (= 204), or, centesimally, 4·9, 23·2, and 71·8, are the proportions of the loss on roasting which are due to the presence of those three substances respectively. Suppose, now, 100 grains of such a mixed ore had been assayed, and by roasting a loss of 40 grains had occurred, then 71·8 per cent. of that loss, or 28·7 grains, was due to arsenical pyrites. But since a loss of 100 in the weight of arsenical pyrites implies the formation of 119 of white arsenic, it follows that, in the case before us, something more than 34 per cent. of that product could be derived from the ore.

We now proceed to describe the metallurgical process. This consists of a roasting or calcination, followed by a resublimation or refining.

1. *Roasting or calcination.*—This is done in one of five different ways; that is to say, either (*a*) in a reverberatory furnace, (*b*) in a Brunton's calciner, (*c*) in an Oxland and Hocking's calciner, (*d*) in kilns, or (*e*) in a muffle-furnace.

(*a*). *Roasting in a Reverberatory Furnace.* — For this there are two kinds of furnace in use. One is a small reverberatory furnace, whose description in Pryce's "Mineralogia Cornubiensis" of last century holds good, with very little change, at the present time.† The flat bed is about 10 feet in length and 3 feet wide towards the fireplace, 5 or 6 feet at the belly, and 18 inches at the flue end, where there is an aperture with an iron door, through which the ore can be stirred by raking. This furnace is applied to the roasting of the tin ores after stamping, when they contain pyrites of any sort. The ore is first dried on the top of the furnace, and then introduced to the quantity of about 8 cwts. and spread equally over the bed; after once heating to a dull red heat it has to be continually turned over with a rake, until all the sulphur and arsenic has been expelled (a process which takes about ten hours), after which the ore is drawn through an aperture in the bed, which is ordinarily closed by an iron plate, the arsenic meanwhile having been deposited in the form of arsenious acid in the flues or chambers, which will in due course be described.

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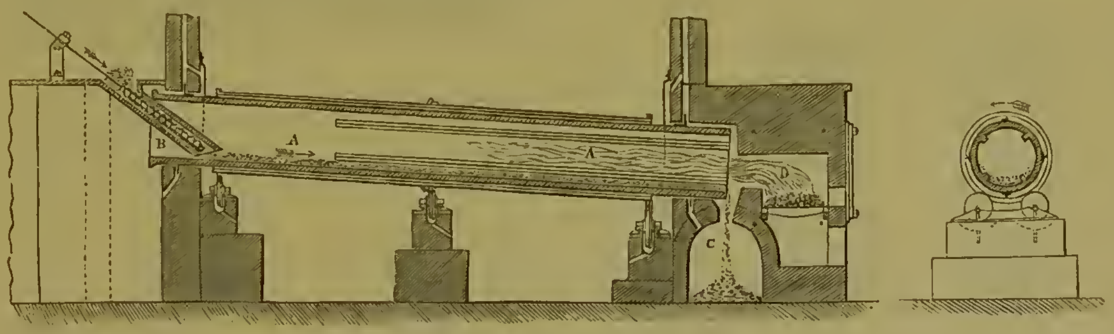
* These numbers are calculated on the supposition that all the copper will have been converted to protoxide, and all the iron to peroxide.

† The furnace, in its present form, is figured in The Proceedings of the Institute of Civil Engineers, vol. vii., Plate 6, figs. 21 and 22, in illustration of Mr. Henderson's paper on the Dressing of Tin and Copper Ores.

The other form of reverberatory furnace is one that is coming increasingly into use, especially where the works are on a large scale. It is generally arranged so as to make a double furnace, or two furnaces put side by side and separated by a wall, while at the end their flues unite; each of them is nearly 30 feet long and 6 or 7 feet wide, with an *inclined bed* sloping gently from the flue towards the fireplace; the fireplace itself is narrow. A great expenditure of fuel is not required, if the ore has much pyrites of any sort, to raise it to the necessary steady, gentle, red heat—the sulphur and arsenic themselves burning keep up the heat; there are seven doors on each side of the double furnace. In the first instance the ore is spread over the bed (to a thickness of 5 or 6 inches) by being thrown in through these seven openings; afterwards it is supplied only through the top door, and the charge is then gradually, as calcination goes on, worked downwards by long levers with a narrow paddle-head. During the time taken by the passage of it along the whole length of the furnace the ore becomes well calcined, when it falls through a slit at the lower end, while other ore is being constantly supplied at the top, and there is a movement of the whole kept up at intervals by the men who *paddle* it down. The finely-stamped tin ore is not thus treated; such a furnace as this is intended for crushed ores containing arsenic, with either copper or perhaps some tin, for which the residue from this process may be afterwards stamped. Yet another form of furnace has been tried, but not with the best results; in this the flames are made to pass *beneath the bed* of the furnace (so as to heat the ore from below) before coming to the bridge, over which, passing, they play as usual on the upper surface of the charge.

Fig 3.



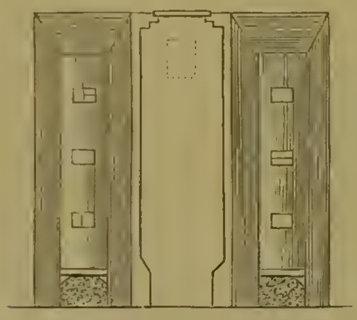
(*b*). *Brunton's Calciner.*—This machine will be understood from the section in Plate I. It may be described as a reverberatory furnace of which the bed revolves; there are, however, *two* fireplaces, the flames from each of which traverse half the circle, and unite at the flue opposite. The revolving bed (which has a diameter of about 8 feet) is of fire-bricks, resting on an iron table; it is higher in the centre than at the circumference; the ore (which for this calciner is the finely-stamped sort) is introduced in the centre through a hopper from a chamber above, where, over the furnace, it had been spread to dry. Fixed in the top of the furnace, and reaching down nearly to the bed, project numerous iron scrapers, shaped like the coulter of a plough, placed obliquely to the radii. On the ore being brought against these in the revolutions of the bed, it is turned over and turned outwards, so that gradually it finds its way down to the circumference, being roasted on the journey, when it falls into a chamber beneath. The bed is made to revolve by steam or water power at the rate of once in about a quarter of an hour; in the figure a water wheel to drive it is shown; the machine is almost self-acting. This calciner is now a good deal used in Cornwall.

(*c*). *Oxland and Hocking's Patent Calciner.*—(Fig. 3.) The principle of this is probably superior to that of the last, but both are liked in Cornwall, and each may be best suited for certain ores. The illustration (which is taken from the "Proceedings of the Institution of Mechanical Engineers," 1873) gives longitudinal and transverse sections. It should be premised that this machine also is intended only for finely-stamped ores. A wrought-iron cylinder, A A, of which the dimensions, as shown in the drawing, are 32 feet in length and 4 feet diameter, is lined with 4 inches of fire-brick, while four longitudinal ribs of fire-brick occur for part of the length. This cylinder is mounted in an inclined position, and made to revolve slowly by machinery (once in eight or ten minutes) upon three, or sometimes only two, pairs of friction wheels. A fire-place, D, is arranged at the lower end, of which the cylinder becomes the flue. The ore is introduced at the top either by an Archimedean screw, B, or from a hopper by hand; in the revolution of the tube it becomes lifted to a certain height, and then falls in such fashion that the particles become well exposed to the oxidizing influence of the flame and air that pass through. In each turn it comes a little lower down the tube, and ultimately reaches the lower end, whence it falls into a chamber, C, in a calcined state, the sulphur and arsenic having been driven off to the flue chambers.

In this calciner as much as 6 or 7 tons of ore can be treated in twenty-four hours. For such a size as we have spoken of the cylinder is not uncommonly made of an old tube of a Cornish boiler; larger ones, however, have been tried, and have done well. The boiler-shell itself may be used for the purpose, the diameter of which would be 5 feet 6 inches or so; in this case the fire-brick lining could be 9 inches thick, so as to leave 4 feet clear inside. It has been found that it is not necessary (at all events with the large ones) to give any inclination to the cylinder; the slope which the ore acquires on being fed from the top is sufficient to carry it down the length of the tube in its revolutions.

(*d*). *Calcining in Kilns.*—For certain kinds of ore this is probably the cheapest method of any. If the ore can be mixed so as to contain something like 20 per cent. of pyrites, the only expenditure of fuel will be at first lighting. The kilns are of the simplest construction. Fig. 4 shows three side by side, of which the middle one is seen in section. The diameter is 3 feet, the height 10 feet. The top is covered with an iron plate, which is removed when the ore is to be introduced, while the flues (one of which is represented by dotted lines) lead away from just beneath it and then unite. At first starting the kiln is filled nearly to the top with rubbish; above is put ore with a little fuel; as this gets hot and begins to burn of itself, some of the rubbish is raked out from below, while more ore (without fuel) is added above. In the course of time all the rubbish will have been got rid of, and the kiln will be filled with a burning mass of ore. Some is then raked from the bottom five or six times in twenty-four hours, and a corresponding amount of ore is put in at top; in this way from 1 ton to 1¼ ton is done per day per kiln, the work being continuously kept up. There will be observed in the kilns three holes (closed up by bricks) at different levels; these are to give access to the ore in case poking should be required to make it descend. With the poorer ores a proportion of fuel may be mixed to produce the necessary heat.

Fig 4.



For this method of roasting the ore may be crushed so as to pass through 1¾-inch meshes. We would point out that, for ores containing tin and a proper proportion of pyrites, it might be advisable to roast them thus and afterwards send them to the stamps, for which they would have been well prepared by the calcination.

(*e*). *Roasting in a Muffle Furnace.*—This is practised at Altenberg and at Reichenstein in Silesia. Figs. 5 and 6 are explanatory of the furnace; the first shows a longitudinal and the second a transverse section; the same characters in each indicate the same parts. The muffle of the furnace, where the ore is deposited for roasting, is indicated by *a*; *b* is a door in the front of the muffle, through which the workman introduces his slicer to work the charge and draw out the exhausted material; *c c c* are the flues round the muffle, where the smoke and flame from the fire are carried forward to the main flue, *e*, leading to the chimney. From the muffle two channels, *f f*, lead to the first chamber, B, appropriated for the condensation of the acid, the further course of which will be spoken of below. For this process the ore is reduced to a moderate size by crushing, in which state it is called *schliech*. About 10 cwts. of the *schliech* forms the charge, which is introduced through the opening, *d*, and spread upon the inclined bed of the furnace to the depth of from 2 to 4 inches; this is raised to a gentle red heat, which is afterwards slightly lowered, and then maintained equably, the ore being at the same time frequently stirred. The door, *b*, is kept open, that the air entering may oxidize the sulphur and arsenic. In about twelve hours this oxidation is effected; the exhausted ore is then raked out and another charge introduced as before.

Fig 5.

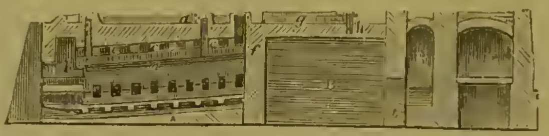
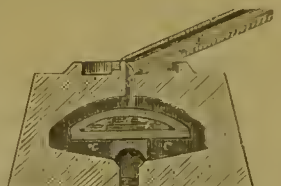


Fig. 6.



The Flues or Chambers.—By whichever of these processes (except it be the last mentioned) the ore may be roasted, the arsenious acid that may be volatilized is intercepted in chambers constructed in the course of the flue, through which all the products of combustion pass between the furnace and the stack. The best arrangement of flue-chambers is that shown in the accompanying diagrams (Figs. 7 and 8). There is a long line of chambers so arranged that the smoke shall zig-zag through them; each chamber is 3 feet wide, about 15 feet long, and 6 feet 6 inches high to the top of the arch. To every two chambers is a low doorway from outside, which is closed by an

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